

trite nitrogen and nitrate nitrogen. Nitrification does not remove significant amounts of nitrogen from the effluent; it only converts nitrogen into another chemical form. Nitrification can be achieved in many suspended and attached growth treatment processes when the processes are designed to foster the growth of nitrifying bacteria. In the traditional activated sludge process, this is accomplished by designing the process to operate at a solids retention time (SRT) that is long enough to prevent slow-growing nitrifying bacteria from being wasted out of the system. Nitrification will also occur in trickling filters that operate at low BOD/TKN ratios either in combination with BOD removal, or as a separate advanced treatment process following any type of secondary treatment. A well-designed and -operated nitrification process will produce an effluent containing 1.0 mg/l or less of ammonia nitrogen.

- **Denitrification** – Denitrification is any wastewater treatment method that completely removes total nitrogen. As with ammonia removal, denitrification is usually best achieved biologically, in which case it must be preceded by nitrification. In biological denitrification, nitrate nitrogen is used by a variety of heterotrophic bacteria as the terminal electron acceptor in the absence of dissolved oxygen. In the process, the nitrate nitrogen is converted to nitrogen gas, which escapes to the atmosphere. The bacteria in these processes also require a carbonaceous food source. Denitrification can be achieved using many alternative treatment processes including variations of many common suspended growth and some attached growth treatment processes, provided that the processes are designed to create the proper microbial environment. Biological denitrification processes can be designed to achieve effluent nitrogen concentrations between 2.0 and 12 mg/l of nitrate nitrogen.
- **Phosphorus Removal** – Phosphorus can be removed from wastewater through chemical or biological methods, or a combination. The choice of methods will depend on site-specific conditions, including the amount of phosphorus to be removed and the desired effluent phosphorus concentration. Chemical phosphorus removal is achieved by precipitating the phosphorus from solution through the addition of iron, aluminum, or calcium salts. Biological phosphorus removal relies on the culturing of bacteria that will store excess amounts of phosphorus when exposed to anaerobic conditions, followed by aerobic conditions in the treatment process. In both cases, the phosphorus is removed from the treatment process with the waste sludge. Chemical phosphorus removal can attain effluent orthophosphorus concentrations
- of less than 0.1 mg/l, while biological phosphorus removal will usually produce an effluent phosphorus concentration between 1.0 and 2.0 mg/l.
- **Coagulation-Sedimentation** – Chemical coagulation with lime, alum, or ferric chloride followed by sedimentation removes SS, heavy metals, trace substances, phosphorus, and turbidity.
- **Carbon Adsorption** – One effective advanced wastewater treatment process for removing biodegradable and refractory organic constituents is granular activated carbon (GAC). Carbon adsorption can reduce the levels of synthetic organic chemicals in secondary effluent by 75 to 85 percent. The basic mechanism of removal is by adsorption of the organic compounds onto the carbon. Carbon adsorption preceded by conventional secondary treatment and filtration can produce an effluent with a BOD of 0.1 to 5.0 mg/l, a COD of 3 to 25 mg/l, and a TOC of 1 to 6 mg/l. Carbon adsorption treatment will also remove several metal ions, particularly cadmium, hexavalent chromium, silver, and selenium. Activated carbon has been used to remove uncharged species, such as arsenic and antimony, from an acidic stream. Carbon adsorption has also been reported as an effective means of removing endocrine disrupting compounds (Hunter and Long, 2002).
- **Membrane Processes** – In recent years, the same factors that favor the use of membranes for potable water treatment (increasing demand, decreasing source water quality, and more stringent regulatory standards) are influencing their use in treating wastewaters prior to reuse. Improvements in membrane technologies which separate suspended solids, dissolved compounds, and human pathogens (protozoan cysts, bacteria and viruses) from reclaimed water have inspired greater confidence in the use of reclaimed water for purposes which include both direct and indirect human contact.

Membrane filters became commercially available in 1927 from the Sartorius Company in Germany. Until the mid-1940s, these filters were used primarily to remove microorganisms and particles from air and water. The first viable reverse osmosis membrane was developed in 1960 by researchers at the University of California at Los Angeles (UCLA). The first commercial reverse osmosis (RO) treatment plant went into service in 1965 in Coalinga, California. The use of membrane filtration systems was initially limited to specialized applications including industrial separation processes and seawater desalination. By

the 1980s, membrane technology was well established.

For many years, membranes were not used for wastewater treatment due to rapid fouling. Prior to 1990, there were a few notable exceptions, including a highly publicized 5-mgd RO system at the Water Factory 21 reclamation plant in Orange County, California. This system went into service in 1975. The plant used cellulose acetate membranes with lime clarification and multi-media filtration for pretreatment prior to the RO system. Another notable exception was a 3.3-mgd ($12 \times 10^3\text{-m}^3/\text{d}$) Petromin plant in Riyadh, Saudi Arabia.

The large-scale use of membranes for wastewater reclamation did not become feasible until the 1980s, when the Australian firm, Memtec, developed a hollow fiber microfiltration membrane system with an air backwash that could provide sustainable operation for wastewater. The Orange County Water District (California) began pilot testing in 1992 to investigate this new microfiltration system as pretreatment for reverse osmosis. The use of this new microfiltration system, followed by thin film composite RO membranes, proved to be a tremendous improvement over the then-conventional system of lime clarification, sand filtration, and cellulose acetate membranes. Between 1994 and 2000, over half a dozen new dual membrane water reclamation systems were constructed in California and Arizona.

Pressure-driven membrane treatment systems are broadly categorized by the size particles rejected by the membrane, or by the molecular weight cut off (MWCO). These classifications include:

Microfiltration (MF)	0.1 μm or 500,000 MWCO
Ultrafiltration (UF)	0.01 μm or 20,000 MWCO
Nanofiltration (NF)	0.001 μm or 200 MWCO
Reverse Osmosis (RO)	0.0001 μm or < 100 MWCO

Figure 3-11 shows a particle size separation comparison chart for conventional filtration, microfiltration, ultrafiltration, and reverse osmosis. **Tables 3-13a** and **3-13b** contain microfiltration and reverse osmosis removal data (Metcalf and Eddy, 2002).

MF systems are used to remove relatively large suspended particles including particulates, large colloids, and oil. This includes providing about 3 to 6 log (99.9 percent to 99.9999 percent) removal of bacteria. In wastewater treatment, MF systems can be used to replace secondary clarifiers and more conventional

(sand) filters following biological treatment. UF membranes have smaller pore sizes than MF membranes and will provide complete removal of bacteria and protozoan cysts, and 4 to 6 log removal for viruses. Otherwise, UF membranes perform the same basic functions in wastewater applications as MF membranes. NF and RO, while retaining smaller particles including molecules and ions, require higher driving pressures, higher levels of pretreatment (prefiltration), and typically operate at lower recovery rates.

For wastewater treatment, the main emphasis has been on MF, UF, and RO membranes. MF and UF have the ability to remove biological contaminants (e.g., bacteria and viruses), and to reduce fouling on downstream reverse osmosis membranes. NF or RO systems are needed where the removal of colloidal and/or dissolved materials is required.

Membrane Bioreactors (MBRs)

MBRs typically consist of UF or MF membranes. These membranes are used to replace conventional gravity clarifiers, and return activated sludge systems in conventional activated sludge biological treatment systems. The membranes can be immersed directly into the aeration tanks, or the mixed liquor can be pumped to external pressure-driven membrane units. MBRs exhibit a number of unique advantages:

- Sludge settling characteristics no longer affect final effluent quality. Biological processes can be operated at much higher suspended solids concentrations and thereby provide greater treatment capacity per unit volume.
- MF and UF membranes provide nearly complete removal of protozoan cysts, suspended solids, and bacteria, as well as partial removal of viruses. In addition to removing suspended solids, UF membranes can retain large organic molecules, improving the biodegradation of otherwise resistant compounds such as grease or emulsified oils.
- Longer sludge ages (as long as 30 to 45 days) are possible, improving the biodegradation of resistant compounds and improving nitrification performance under adverse conditions (such as low temperature).
- Wasting occurs directly from the aeration basin, improving process control.
- Submerged MBR systems are well suited to upgrade existing systems with minimum new construction required and low impact to ongoing operations.

Table 3-13a. Microfiltration Removal Performance Data

Constituent	MF Influent (mg/l)	MF Effluent (mg/l)	Average Reduction (%)	Reduction Reported in Literature (%)
TOC	10-31	9-16	57	45-65
BOD	11-32	<2-9.9	86	75-90
COD	24-150	16-53	76	70-85
TSS	8-46	<0.5	97	95-98
TDS	498-622	498-622	0	0-2
NH ₃ -N	21-42	20-35	7	5-15
NO ₃ -N	<1-5	<1-5	0	0-2
PO ₄ ⁻	6-8	6-8	0	0-2
SO ₄ ²⁻	90-120	90-120	0	0-1
Cl ⁻	93-115	93-115	0	0-1
Turbidity	2-50 NTU	0.03-0.08 NTU	>99	---

¹ Data collected from the Dublin San Ramon Sanitary District for the period from April 2000 through December, 2000.

² Typical flux rate during test period was 1600 l/m²-d.

Adapted from: Metcalf and Eddy, 2002

Table 3-13b. Reverse Osmosis Performance Data

Constituent	RO Influent (mg/l)	RO Effluent (mg/l)	Average Reduction (%)	Reduction Reported in Literature (%)
TOC	9-16	<0.5	>94	85-95
BOD	<2-9.9	<2	>40	30-60
COD	16-53	<2	>91	85-95
TSS	<0.5	~0	>99	95-100
TDS	498-622	9-19	---	90-98
NH ₃ -N	20-35	1-3	96	90-98
NO ₃ -N	<1-5	0.08-3.2	96	65-85
PO ₄ ⁻	8-Jun	0.1-1	~99	95-99
SO ₄ ²⁻	90-120	<0.5-0.7	99	95-99
Cl ⁻	93-115	0.9-5.0	97	90-98
Turbidity	0.03-0.08 NTU	0.03 NTU	50	40-80

¹ Data collected from the Dublin San Ramon Sanitary District for the period from April 1999 through December, 1999.

² Typical flux rate during test period was 348 l/m²-d.

Adapted from: Metcalf and Eddy, 2002

Submerged membrane assemblies, either MF or UF, are typically composed of bundles of hollow fiber or flat sheets of microporous membranes. Filtrate is drawn through the membrane assemblies by means of a vacuum applied to the product side of the mem-

brane. Turbulence on the exterior (feed side) is maintained by diffused aeration to reduce fouling.

Low-pressure membrane filtration (MF or UF) can be used following secondary clarification to provide a

higher degree of solids removal. Operating in a conventional (pressurized) flow pattern, clarified effluent is further treated to remove particulate material (MF) or colloidal material (UF). Typical operating pressures range from 20 to 100 psi (100 to 700 KPa), and reject flows range from 2 to 50 percent. MF and UF membranes can be used to pre-treat flow prior to NF or RO treatment.

Higher-pressure NF and RO systems are used to remove dissolved organic and inorganic compounds. The smaller pore size (lower MWCO) results in higher quality product water, which may meet primary and secondary drinking water standards. The higher rates of rejection also result in increasing problems for disposing of the concentrate streams.

- Other Processes — Other advanced wastewater treatment processes of constituent removal include ammonia stripping, breakpoint chlorination for ammonia removal, and selective ion exchange for nitrogen removal.

3.4.3 Reliability in Treatment

A high standard of reliability, similar to water treatment plants, is required at wastewater reclamation plants. Because there is potential for harm (i.e., in the event that improperly treated reclaimed water is delivered to the use area), water reuse requires strict conformance to all applicable water quality parameters. The need for reclamation facilities to reliably and consistently produce and distribute reclaimed water of adequate quality and quantity is essential and dictates that careful attention be given to reliability features during the design, construction, and operation of the facilities.

A number of fallible elements combine to make up an operating water reclamation system. These include the power supply, individual treatment units, mechanical equipment, the maintenance program, and the operating personnel. An array of design features and non-design provisions can be employed to improve the reliability of the separate elements and the system as a whole. Back-up systems are important in maintaining reliability in the event of failure of vital components. Particularly critical units include the disinfection system, power supply, and various treatment unit processes.

For reclaimed water production, EPA Class I reliability is recommended as a minimum criteria. Class I reliability requires redundant facilities to prevent treatment upsets during power and equipment failures, flooding, peak loads, and maintenance shutdowns. Reliability for water reuse should also consider:

- Operator certification to ensure that qualified personnel operate the water reclamation and reclaimed water distribution systems
- Instrumentation and control systems for on-line monitoring of treatment process performance and alarms for process malfunctions
- A comprehensive quality assurance program to ensure accurate sampling and laboratory analysis protocol
- Adequate emergency storage to retain reclaimed water of unacceptable quality for re-treatment or alternative disposal
- Supplemental storage and/or water supply to ensure that the supply can match user demands
- A strict industrial pretreatment program and strong enforcement of sewer use ordinances to prevent illicit dumping into the collection system of hazardous materials or other materials that may interfere with the intended use of the reclaimed water
- A comprehensive operating protocol that defines the responsibilities and duties of the operations staff to ensure the reliable production and delivery of reclaimed water

Many states have incorporated procedures and practices into their reuse rules and guidelines to enhance the reliability of reclaimed water systems. Florida requires the producer of reclaimed water to develop a detailed operating protocol for all public access systems. This protocol must identify critical monitoring and control equipment, set points for chlorine and turbidity, actions to be taken in the event of a failure to achieve these limits, and procedures to clear the substandard water and return to normal operations (FAC 62-610). Washington is in the process of developing Water Reclamation Facilities Reliability Assessment Guidance, which includes an alarm and reliability checklist.

3.4.3.1 EPA Guidelines for Reliability

More than 30 years ago, before the Federal Water Quality Administration evolved into the EPA, it recognized the importance of treatment reliability, issuing guidelines entitled, "Federal Guidelines: Design, Operation and Maintenance of Waste Water Treatment Facilities" (Federal Water Quality Administration, 1970). These guidelines provided an identification and description of various reliability provisions and included the following concepts or principles regarding treatment plant reliability:

Table 4-11. Groundwater Recharge ⁽¹⁾

	Arizona	California ⁽²⁾	Florida	Hawaii	Nevada	Texas	Washington
Treatment	NR ⁽³⁾	Case-by-case basis	Secondary treatment and basic disinfection	Case-by-case basis	NR	NR	Oxidized, coagulated, filtered, and disinfected
BOD₅	NR		NS ⁽⁴⁾		NR	NR	5 mg/l
TSS	NR		10.0 mg/l		NR	NR	5 mg/l
Turbidity	NR		NS		NR	NR	2 NTU (Avg) 5 NTU (Max)
Coliform	NR		NS		NR	NR	Total 2.2/100 ml (Avg) 23/100 ml (Max)
Total Nitrogen	NR		12 mg/l		NR	NR	NS

- (1) All state requirements are for groundwater recharge via rapid-rate application systems. Additional regulations for recharge of potable aquifers are contained in Section 4.1.1.10 and Appendix A.
- (2) Groundwater recharge in California and Hawaii is determined on a case-by-case basis
- (3) NR - Not regulated by the state
- (4) NS - Not specified by state regulations

ter defined as oxidized, coagulated, filtered, and disinfected. Total coliform is not to exceed 2.2/100 ml as a 7-day median and 23/100 ml in any sample. Weekly average BOD and TSS limits are set at 5 mg/l. Turbidity is not to exceed 2 NTU as a monthly average and 5 NTU in any sample. Additionally, groundwater monitoring is required and is based on reclaimed water quality and quantity, site-specific soil and hydrogeologic characteristics, and other considerations. Washington also specifies that reclaimed water withdrawn for nonpotable purposes can be withdrawn at any distance from the point of injection and at any time after direct recharge.

Florida requires that TSS not exceed 5.0 mg/l in any sample, be achieved prior to disinfection, and that the total nitrogen in the reclaimed water be less than 12 mg/l. Florida also requires continuous on-line monitoring of turbidity; however, no limit is specified.

4.1.1.10 Indirect Potable Reuse

Indirect potable reuse involves the use of reclaimed water to augment surface water sources that are used or will be used for public water supplies or to recharge groundwater used as a source of domestic water supply. Unplanned indirect potable water reuse is occurring in many

river systems today. Many domestic wastewater treatment plants discharge treated effluent to surface waters upstream of intakes for domestic water supply treatment plants. Additionally, many types of beneficial reuse projects inadvertently contribute to groundwater augmentation as an unintended result of the primary activity. For example, irrigation can replenish groundwater sources that will eventually be withdrawn for use as a potable water supply. Indirect potable reuse systems, as defined here, are distinguished from typical groundwater recharge systems and surface water discharges by both intent and proximity to subsequent withdrawal points for potable water use. Indirect potable reuse involves the intentional introduction of reclaimed water into the raw water supply for the purposes of increasing the total volume of water available for potable use. In order to accomplish this objective, the point at which reclaimed water is introduced into the environment must be selected to ensure it will flow to the point of withdrawal. Typically the design of these systems assumes there will be little to no additional treatment in the environment after discharge, and all applicable water quality requirements are met prior to release of the reclaimed water.

Based on a review of the existing reuse regulations and guidelines, 4 of the 7 states (California, Florida, Hawaii,

and Washington) have regulations or guidelines pertaining to indirect potable reuse. For groundwater recharge of potable aquifers, most of the states require a pretreatment program, public hearing requirements prior to project approval, and a groundwater monitoring program. Florida and Washington require pilot plant studies to be performed. In general, all the states that specify treatment processes require secondary treatment with filtration and disinfection. Washington is the only state that specifies the wastewater must be treated by reverse osmosis. California and Hawaii do not specify the type of treatment processes required and determine requirements on a case-by-case basis.

Most states specify reclaimed water quality limitations for TSS, nitrogen, total organic carbon (TOC), turbidity, and total coliform. Florida requires that TSS not exceed 5.0 mg/l in any sample and be achieved prior to disinfection. Florida and Washington require the total nitrogen in the reclaimed water to be less than 10 mg/l. Washington has a limit of 1 mg/l for TOC, while Florida's limit is set at 3 mg/l as a monthly average. Florida also requires an average limit of 0.2 mg/l for total organic halides (TOX). Turbidity limits vary greatly where specified. For example, Washington specifies a limit of 0.1 NTU as a monthly average and 0.5 NTU as a maximum at any time. Florida requires continuous on-line monitoring of turbidity; however, no limit is specified. Fecal coliform limits also vary greatly from state to state. Washington requires a limit of 1/100 ml for total coliform as a weekly median and a not to exceed limit of 5/100 ml in any one sample for direct injection into a potable aquifer. The states that specify reclaimed water quality limitations require the reclaimed water to meet drinking water standards.

Most states specify a minimum time the reclaimed water must be retained underground prior to being withdrawn as a source of drinking water. Washington requires that reclaimed water be retained underground for a minimum of 12 months prior to being withdrawn as a drinking water supply. Several states also specify minimum separation distances between a point of recharge and the point of withdrawal as a source of drinking water. Florida requires a 500-foot (150-meter) separation distance between the zone of discharge and potable water supply well. Washington requires the minimum horizontal separation distance between the point of direct recharge and point of withdrawal as a source of drinking water supply to be 2,000 feet (610 meters). **Table 4-12** shows the reclaimed water quality and treatment requirements for indirect potable reuse.

Florida includes discharges to Class I surface waters (public water supplies) as indirect potable reuse. Discharges less than 24 hours travel time upstream from

Class I waters are also considered as indirect potable reuse. Surface water discharges located more than 24 hours travel time to Class I waters are not considered indirect potable reuse. For discharge to Class I surface waters or water contiguous to or tributary to Class I waters (defined as a discharge located less than or equal to 4 hours travel time from the point of discharge to arrival at the boundary of the Class I water), secondary treatment with filtration, high-level disinfection, and any additional treatment required to meet TOC and TOX limits is required. The reclaimed water must meet primary and secondary drinking water standards, except for asbestos, prior to discharge. TSS must not exceed 5.0 mg/l in any sample prior to disinfection and total nitrogen cannot exceed 10 mg/l as an annual average. The reclaimed water must also meet TOC limitations of 3 mg/l as a monthly average and 5 mg/l in any single sample. Outfalls for surface water discharges are not to be located within 500 feet (150 meters) of existing or approved potable water intakes within Class I surface waters.

4.1.2 Reclaimed Water Monitoring Requirements

Reclaimed water monitoring requirements vary greatly from state to state and again depend on the type of reuse. For unrestricted urban reuse, Oregon requires sampling for coliform daily, while for agricultural reuse of non-food crops, sampling for total coliform is only required once a week. Oregon also requires hourly monitoring of turbidity when a limit on turbidity is specified.

For unrestricted and restricted urban reuse, as well as agricultural reuse on food crops, Florida requires the continuous on-line monitoring of turbidity and chlorine residual. Even though no limits on turbidity are specified in Florida, continuous monitoring serves as an on-line surrogate for suspended solids. In addition, Florida requires that the TSS limit be achieved prior to disinfection and has a minimum schedule for sampling and testing flow, pH, chlorine residual, dissolved oxygen, TSS, CBOD, nutrients, and fecal coliform based on system capacity. Florida also requires an annual analysis of primary and secondary drinking water standards for reclaimed water used in irrigation for facilities greater than 100,000 gpd (4.4 l/s). Monitoring for *Giardia* and *Cryptosporidium* must also be performed with frequency dependent on system capacity. Other states determine monitoring requirements on a case-by-case basis depending on the type of reuse.

4.1.3 Treatment Facility Reliability

Some states have adopted facility reliability regulations or guidelines in place of, or in addition to, water quality

Table 4-12. Indirect Potable Reuse ⁽¹⁾

	Arizona	California ⁽²⁾	Florida	Hawaii	Nevada	Texas	Washington
Treatment	NR ⁽³⁾	Case-by-case basis	Advanced treatment, filtration, and high-level disinfection	Case-by-case basis	NR	NR	Oxidized, coagulated, filtered, reverse-osmosis treated, and disinfected
BOD ₅	NR		20 mg/l		NR	NR	5 mg/l
TSS	NR		5.0 mg/l		NR	NR	5 mg/l
Turbidity	NR		NS ⁽⁴⁾		NR	NR	0.1 NTU (Avg) 0.5 NTU (Max)
Coliform	NR		Total		NR	NR	Total
			All samples less than detection				1/100 ml (Avg)
							5/100 ml (Max)
Total Nitrogen	NR		10 mg/l		NR	NR	10 mg/l
TOC	NR		3 mg/l (Avg)		NR	NR	1.0 mg/l
			5 mg/l (Max)				
Primary and Secondary Standards	NR		Compliance with most primary and secondary		NR	NR	Compliance with most primary and secondary

(1) Florida requirements are for the planned use of reclaimed water to augment surface water sources that will be used as a source of domestic water supply

(2) Indirect potable reuse in California and Hawaii is determined on a case-by-case basis

(3) NR - Not regulated by the state

(4) NS - Not specified by state regulations

requirements. Generally, requirements consist of alarms warning of power failure or failure of essential unit processes, automatic standby power sources, emergency storage, and the provision that each treatment process be equipped with multiple units or a back-up unit.

Articles 8, 9, and 10 of California's Title 22 regulations provide design and operational considerations covering alarms, power supply, emergency storage and disposal, treatment processes, and chemical supply, storage, and feed facilities. For treatment processes, a variety of reliability features are acceptable in California. For example, for all biological treatment processes, one of the following is required:

- Alarm (failure and power loss) and multiple units capable of producing biologically oxidized wastewater with one unit not in operation

- Alarm (failure and power loss) and short-term (24-hour) storage or disposal provisions and standby replacement equipment

- Alarm (failure and power loss) and long-term (20-day) storage or disposal provisions

Florida requires Class I reliability of treatment facilities when reclaimed water is used for irrigation of food crops and for restricted and unrestricted urban reuse. Class I reliability requires multiple treatment units or back-up units and a secondary power source. In addition, a minimum of 1 day of reject water storage is required to store reclaimed water of unacceptable quality for additional treatment. Florida also requires staffing at the water reclamation facility 24 hours/day, 7 days/week or 6 hours/day, 7 days/week. The minimum staffing requirement may be reduced to 6 hours/day, 7 days/week if reclaimed water

is delivered to the reuse system only during periods when a qualified operator is present, or if additional reliability features are provided.

Florida has also established minimum system sizes for treatment facilities to aid in assuring the continuous production of high-quality reclaimed water. Minimum system size for unrestricted and restricted urban reuse and for use on edible crops is 0.1 mgd (4.4 l/s). A minimum system size is not required if reclaimed water will be used only for toilet flushing and fire protection uses.

Other states that have regulations or guidelines regarding treatment facility reliability include Georgia, Hawaii, Indiana, Massachusetts, North Carolina, Oregon, Utah, Washington, and Wyoming. Washington's guidelines pertaining to treatment facility reliability are similar to California's regulations. Georgia, Massachusetts, North Carolina, Oregon, and Wyoming require that multiple treatment units be provided for all essential treatment processes and a secondary or back-up power source be supplied.

4.1.4 Reclaimed Water Storage

Current regulations and guidelines regarding storage requirements are primarily based upon the need to limit or prevent surface water discharge and are not related to storage required to meet diurnal or seasonal variations in supply and demand. Storage requirements vary from state to state and are generally dependent upon geographic location and site conditions. For example, Florida requires a minimum storage volume equal to 3 days of the average design flow, while South Dakota requires a minimum storage volume of 210 days of the average design flow. The large difference in time is primarily due to the high number of non-irrigation days due to freezing temperatures in the northern states. In addition to the minimum storage requirement, Florida also requires that a water balance be performed based on a 1-in-10 year rainfall recurrence interval and a minimum of 20 years of climatic data to determine if additional storage is required beyond the minimum requirement of 3 days.

Most states that specify storage requirements do not differentiate between operational and seasonal storage, with the exception of Delaware, Georgia, and Ohio, which require that both operational and wet weather storage be considered. The majority of states that have storage requirements in their regulations or guidelines require that a water balance be performed on the reuse system, taking into account all inputs and outputs of water to the system based on a specified rainfall recurrence interval.

Presently, Florida is the only state with regulations or guidelines for aquifer storage and recovery (ASR) of reclaimed water. ASR systems using reclaimed water are required to meet the technical and permitting requirements of Florida's Department of Environmental Protection underground injection control program and obtain an underground injection control construction and operation permit in addition to the domestic wastewater permit. Water recovered from the ASR system must meet the performance standards for fecal coliform as specified for high-level disinfection. Specifically, the fecal coliform limits require 75 percent of samples to be below detection limits, and any single sample is not to exceed 25/100 ml before use in a reuse system. Preapplication treatment and disinfection requirements vary depending on the class of groundwater receiving injected reclaimed water, but may be as stringent as to require that reclaimed water meet primary and secondary drinking water standards and TOC and TOX limits prior to injection. Monitoring of the reclaimed water prior to injection and after recovery from the ASR system is required. In addition, a groundwater monitoring plan must be implemented before placing the ASR system into operation. The monitoring plan must be designed to verify compliance with the groundwater standards and to monitor the performance of the ASR system. As part of the monitoring plan, a measure of inorganics concentration (such as chlorides or total dissolved solids) and specific conductance of the water being injected, the groundwater, and the recovered water are required to be monitored. In some cases, an extended zone of discharge for the secondary drinking water standards and for sodium can be approved.

Injection wells and recovery wells used for ASR are to be located at least 500 feet from any potable water supply well. For potable water supply wells that are not public water supply wells, a smaller setback distance may be approved if it can be demonstrated that confinement exists such that the system will not adversely affect the quantity or quality of the water withdrawn from the potable water supply well. If the ASR well is located in the same aquifer as a public supply well, the permitting agencies may require a detailed analysis of the potential for reclaimed water entry into the public supply well.

4.1.5 Application Rates

When regulations specify application or hydraulic loading rates, the regulations generally pertain to land application systems that are used primarily for additional wastewater treatment for disposal rather than reuse. When systems are developed chiefly for the purpose of land treatment and/or disposal, the objective is often to dispose of as much effluent on as little land as possible:

Table 4-13. Suggested Guidelines for Water Reuse ¹

Types of Reuse	Treatment	Reclaimed Water Quality ²	Reclaimed Water Monitoring	Setback Distances ³	Comments
<i>Groundwater Recharge</i> By spreading or injection into aquifers not used for public water supply	<ul style="list-style-type: none"> • Site-specific and use dependent • Primary (minimum) for spreading • Secondary ⁴ (minimum) for injection 	<ul style="list-style-type: none"> • Site-specific and use dependent 	<ul style="list-style-type: none"> • Depends on treatment and use 	<ul style="list-style-type: none"> • Site-specific 	<ul style="list-style-type: none"> • Facility should be designed to ensure that no reclaimed water reaches potable water supply aquifers • See Section 2.5 for more information. • For spreading projects, secondary treatment may be needed to prevent clogging. • For injection projects, filtration and disinfection may be needed to prevent clogging. • See Section 3.4.3 for recommended treatment reliability.
<i>Indirect Potable Reuse</i> Groundwater recharge by spreading into potable aquifers	<ul style="list-style-type: none"> • Secondary ⁴ • Disinfection ⁵ • May also need filtration ⁶ and/or advanced wastewater treatment ¹⁶ 	<ul style="list-style-type: none"> • Secondary ⁴ • Disinfection ⁵ • Meet drinking water standards after percolation through vadose zone 	<ul style="list-style-type: none"> Includes, but not limited to, the following: <ul style="list-style-type: none"> • pH - daily • Coliform - daily • Cl₂ residual - continuous • Drinking water standards - quarterly • Other ¹⁷ - depends on constituent • BOD - weekly • Turbidity - continuous 	<ul style="list-style-type: none"> • 500 ft (150 m) to extraction wells. May vary depending on treatment provided and site-specific conditions. 	<ul style="list-style-type: none"> • The depth to groundwater (i.e., thickness to the vadose zone) should be at least 6 feet (2 m) at the maximum groundwater mounding point. • The reclaimed water should be retained underground for at least 6 months prior to withdrawal. • Recommended treatment is site-specific and depends on factors such as type of soil, percolation rate, thickness of vadose zone, native groundwater quality, and dilution. • Monitoring wells are necessary to detect the influence of the recharge operation on the groundwater. • See Sections 2.5 and 2.6 for more information. • The reclaimed water should not contain measurable levels of viable pathogens after percolation through the vadose zone. ¹² • See Section 3.4.3 for recommended treatment reliability.
<i>Indirect Potable Reuse</i> Groundwater recharge by injection into potable aquifers	<ul style="list-style-type: none"> • Secondary ⁴ • Filtration ⁵ • Disinfection ⁵ • Advanced wastewater treatment ¹⁶ 	<ul style="list-style-type: none"> Includes, but not limited to, the following: <ul style="list-style-type: none"> • pH = 6.5 - 8.5 • ≤ 2 NTU ⁸ • No detectable total col/100 ml ^{9,10} • 1 mg/l Cl₂ residual (minimum) ¹¹ • ≤ 3 mg/l TOC • ≤ 0.2 mg/l TOX • Meet drinking water standards 	<ul style="list-style-type: none"> Includes, but not limited to, the following: <ul style="list-style-type: none"> • pH - daily • Turbidity - continuous • Total coliform - daily • Cl₂ residual - continuous • Drinking water standards - quarterly • Other ¹⁷ - depends on constituent 	<ul style="list-style-type: none"> • 2000 ft (600 m) to extraction wells. May vary depending on site-specific conditions. 	<ul style="list-style-type: none"> • The reclaimed water should be retained underground for at least 9 months prior to withdrawal. • Monitoring wells are necessary to detect the influence of the recharge operation on the groundwater. • Recommended quality limits should be met at the point of injection. • The reclaimed water should not contain measurable levels of viable pathogens after percolation through the vadose zone. ¹² • See Sections 2.5 and 2.6 for more information. • A higher chlorine residual and/or a longer contact time may be necessary to assure virus and protozoa inactivation. • See Section 3.4.3 for recommended treatment reliability.
<i>Indirect Potable Reuse</i> Augmentation of surface supplies	<ul style="list-style-type: none"> • Secondary ⁴ • Filtration ⁵ • Disinfection ⁵ • Advanced wastewater treatment ¹⁶ 	<ul style="list-style-type: none"> Includes, but not limited to, the following: <ul style="list-style-type: none"> • pH = 6.5 - 8.5 • ≤ 2 NTU ⁸ • No detectable total col/100 ml ^{9,10} • 1 mg/l Cl₂ residual (minimum) ¹¹ • ≤ 3 mg/l TOC • Meet drinking water standards 	<ul style="list-style-type: none"> Includes, but not limited to, the following: <ul style="list-style-type: none"> • pH - daily • Turbidity - continuous • Total coliform - daily • Cl₂ residual - continuous • Drinking water standards - quarterly • Other ¹⁷ - depends on constituent 	<ul style="list-style-type: none"> • Site-specific 	<ul style="list-style-type: none"> • Recommended level of treatment is site-specific and depends on factors such as receiving water quality, time and distance to point of withdrawal, dilution and subsequent treatment prior to distribution for potable uses. • The reclaimed water should not contain measurable levels of viable pathogens. ¹² • See Sections 2.6 for more information. • A higher chlorine residual and/or a longer contact time may be necessary to assure virus and protozoa inactivation. • See Section 3.4.3 for recommended treatment reliability.

Footnotes

1. These guidelines are based on water reclamation and reuse practices in the U.S., and they are especially directed at states that have not developed their own regulations or guidelines. While the guidelines should be useful in many areas outside the U.S., local conditions may limit the applicability of the guidelines in some countries (see Chapter 8). It is explicitly stated that the direct application of these suggested guidelines will not be used by USAID as strict criteria for funding.
2. Unless otherwise noted, recommended quality limits apply to the reclaimed water at the point of discharge from the treatment facility.
3. Setback distances are recommended to protect potable water supply sources from contamination and to protect humans from unreasonable health risks due to exposure to reclaimed water.
4. Secondary treatment processes include activated sludge processes, trickling filters, rotating biological contractors, and may include stabilization pond systems. Secondary treatment should produce effluent in which both the BOD and TSS do not exceed 30 mg/l.
5. Filtration means the passing of wastewater through natural undisturbed soils or filter media such as sand and/or anthracite, filter cloth, or the passing of wastewater through microfilters or other membrane processes.
6. Disinfection means the destruction, inactivation, or removal of pathogenic microorganisms by chemical, physical, or biological means. Disinfection may be accomplished by chlorination, UV radiation, ozonation, other chemical disinfectants, membrane processes, or other processes. The use of chlorine as defining the level of disinfection does not preclude the use of other disinfection processes as an acceptable means of providing disinfection for reclaimed water.
7. As determined from the 5-day BOD test.
8. The recommended turbidity limit should be met prior to disinfection. The average turbidity should be based on a 24-hour time period. The turbidity should not exceed 5 NTU at any time. If TSS is used in lieu of turbidity, the TSS should not exceed 5 mg/l.
9. Unless otherwise noted, recommended coliform limits are median values determined from the bacteriological results of the last 7 days for which analyses have been completed. Either the membrane filter or fermentation-tube technique may be used.
10. The number of fecal coliform organisms should not exceed 14/100 ml in any sample.
11. Total chlorine residual should be met after a minimum contact time of 30 minutes.
12. It is advisable to fully characterize the microbiological quality of the reclaimed water prior to implementation of a reuse program.
13. The number of fecal coliform organisms should not exceed 800/100 ml in any sample.
14. Some stabilization pond systems may be able to meet this coliform limit without disinfection.
15. Commercially processed food crops are those that, prior to sale to the public or others, have undergone chemical or physical processing sufficient to destroy pathogens.
16. Advanced wastewater treatment processes include chemical clarification, carbon adsorption, reverse osmosis and other membrane processes, air stripping, ultrafiltration, and ion exchange.
17. Monitoring should include inorganic and organic compounds, or classes of compounds, that are known or suspected to be toxic, carcinogenic, teratogenic, or mutagenic and are not included in the drinking water standards.

CHAPTER 7

Public Involvement Programs

In the years since this manual was first developed, the world has seen ever-increasing demands for water, often from competing interests, and often in the face of declining water supplies. As a result, water quality and quantity have become important public topics in many arenas, and regulatory agencies often require some level of stakeholder involvement in water management decisions. This is strikingly different from the past when members of the public were often informed about projects only after final decisions had been made. Today, responsible leaders recognize the need to incorporate public values with science, technology, and legal aspects to create real, workable solutions tailored to meet specific needs.

In the area of water reuse, the opportunities for meaningful public involvement are many. This chapter provides an overview of the key elements of public planning, as well as several case studies illustrating public involvement and/or participation approaches.

7.1 Why Public Participation?

Public involvement or participation programs work to identify key audiences and specific community issues at a very early stage, offering information and opportunities for input in a clear, understandable way. Effective public involvement begins at the earliest planning stage and lasts through implementation and beyond.

Public participation begins with having a clear understanding of the water reuse options available to the community. Once an understanding of possible alternatives is developed, a list of stakeholders, including possible users, can be identified and early public contacts may begin. Why begin contacting stakeholders before a plan is in place? These citizen stakeholders can provide early indications regarding which reuse program will be best accepted on a community-wide level. Beyond that, informed citizens can help identify and resolve potential problems before they occur and develop alternatives that may work more effectively for the community.

In general, effective public participation programs invite two-way communication, provide education, and ask for meaningful input as the reuse program is developed and refined. Depending on the project, public involvement can involve limited contact with a number of specific users, or can be expanded to include the formation of a formal advisory committee or task force. Often, public information efforts begin by targeting the most impacted stakeholders. Over time, as an early education base is built among stakeholders, the education effort then broadens to include the public at large. Regardless of the audience, all public involvement efforts are geared to help ensure that adoption of a selected water reuse program will fulfill real user needs and generally recognized community goals including public health, safety, and program cost.

The term, "two-way communications flow" cannot be too highly emphasized. In addition to building community support for a reuse program, public participation can also provide valuable community-specific information to the reuse planners. Citizens have legitimate concerns, quite often reflecting their knowledge of detailed technical information. In reuse planning, especially, where one sector of "the public" comprises potential users of reclaimed water, this point is critical. Potential users *generally* know what flow and quality of reclaimed water are acceptable for their applications.

7.1.1 Informed Constituency

By taking time during the planning stages to meet with citizens, communities will have a much greater opportunity to develop a successful reuse program. Many citizens may have a pre-conceived notion about reclaimed water and its benefits. It is important to identify each stakeholder's issues and to address questions and concerns in a clear, matter-of-fact way. This two-way dialogue will lead to informed input regarding reuse alternatives.

A public participation program can build, over time, an informed constituency that is comfortable with the concept of reuse, knowledgeable about the issues involved in reclamation/reuse, and supportive of program implementation. Ideally, citizens who have taken part in the planning process will be effective proponents of the selected plans. Having educated themselves on the issues involved in adopting reclamation and reuse, they will also understand how various interests have been accommodated in the final plan. Their understanding of the decision-making process will, in turn, be communicated to larger interest groups – neighborhood residents, clubs, and municipal agencies – of which they are a part. Indeed the potential reuse customer who is enthusiastic about the prospect of receiving service may become one of the most effective means of generating support for a program. This is certainly true with the urban reuse programs in St. Petersburg and Venice, Florida. In these communities, construction of distribution lines is contingent on the voluntary participation of a percentage of customers within a given area.

In other communities where reuse has not been introduced in any form, the focus may begin with very small, specific audiences. For instance, a community may work closely with golf course owners and superintendents to introduce reuse water as a resource to keep the golf course in prime condition, even at times when other water supplies are low. This small, informed constituency can then provide the community with a lead-in to other reclaimed water options in the future. Golf course superintendents spread the word informally, and, as golfers see the benefits, the earliest of education campaigns has subtly begun. Later, the same community may choose to introduce an urban system, offering reclaimed water for irrigation use.

Since many reuse programs may ultimately require a public referendum to approve a bond issue for funding reuse system capital improvements, diligently soliciting community viewpoints and addressing any concerns early in the planning process can be invaluable in garnering support. Public involvement early in the planning process, even as alternatives are beginning to be identified, allows ample time for the dissemination and acceptance of new ideas among the constituents. Public involvement can even expedite a reuse program by uncovering any opposition early enough to adequately address citizen concerns and perhaps modify the program to better fit the community.

7.2 Defining the “Public”

Many contemporary analyses of public involvement define “the public” as comprising various subsets of “pub-

lics” with differing interests, motivations, and approaches to policy issues. For example, in discussing public participation for wastewater facilities and reuse planning the following publics may be identified: general public, potential users, environmental groups, special interest groups, home owners associations, regulators and/or regulating agencies, educational institutions, political leaders, and business/academic/community leaders. In an agricultural area, there may be another different set of publics including farmers.

For example, several government agencies in California held a Reuse Summit in 1994, at which they endorsed the creation of the public outreach effort by creating the following mission statement (Sheikh *et al.*, 1996):

“To activate community support for water recycling through an outreach program of educating and informing target audiences about the values and benefits of recycled water.”

During that summit they also identified 8 public audiences: Local Elected Officials, Regulatory Agency Staff, General Public, Environmental Community, City Planning Staffs, Agricultural Community, Schools, and Newspaper Editorial Boards.

From the outset of reuse planning, informal consultation with members of each of the groups comprising “the public”, and formal presentations before them, should both support the development of a sound base of local water reuse information and, simultaneously, build a coalition that can effectively advocate reuse in the community. Keeping in mind that different groups have different interests at stake, each presentation should be tailored to the special needs and interests of the audience.

If a reuse program truly has minimal impact on the general public, limited public involvement may be appropriate. For example, use of reclaimed water for industrial cooling and processing – with no significant capital improvements required of the municipality – may require support only from regulatory, technical, and health experts, as well as representatives from the prospective user and its employees. Reuse for pastureland irrigation in isolated areas might be another example warranting only limited public participation.

7.3 Overview of Public Perceptions

One of the most tried and true methods of determining the public’s perception of reuse programs is surveys. Surveys can determine whether or not there will be a large enough consumer base to sustain a program, if the pro-

gram will be favorable enough to progress to the conceptual and design stage, and the overall success of the project after implementation. The following projects highlight different survey strategies and results across the nation.

7.3.1 Residential and Commercial Reuse in Tampa, Florida

A survey done by the City of Tampa for its residential reuse project included a direct mailing and public opinion survey. Information was sent to 15,500 potable water customers in the conceptual project area. Out of the pool of potential reuse customers, 84 percent of the residential users and 94 percent of the commercial users in the South Tampa area thought that reclaimed water was safe for residential and commercial landscape irrigation. Of the same group, 84 percent of the residential responders and 90 percent of the commercial responders replied that the project was appealing. The responses met the design criteria of 90 percent participation (Grosh *et al.*, 2002).

7.3.2 A Survey of WWTP Operators and Managers

A study done by Hall and Rubin in 2002 surveyed 50 wastewater operators and managers. Seventy percent of the responders stated that they believed that reuse would be an important part of their operation in 5 years. The majority (66 percent) thought that water reuse should be considered as an element of all water and wastewater expansion facility permits. Ninety percent wanted funding agencies to consider financial incentives to encourage more water reuse. **Table 7-1** lists the survey results (in percentages) to the inquiry for potential use alternatives for reclaimed water.

7.3.3 Public Opinion in San Francisco, California

The City of San Francisco, California, surveyed the general public to measure public acceptance of a proposed reclaimed water project. **Figures 7-1** and **7-2** graphically demonstrate the responses that were collected. The overall majority strongly felt that reclaimed water was beneficial. **Figure 7-2** shows that the responders felt positively about all of the proposed uses of reclaimed water: fire fighting, irrigation of golf courses and parks, street cleaning, toilet flushing, and drought protection.

7.3.4 Clark County Sanitation District Water Reclamation Opinion Surveys

Clark County (Las Vegas, Nevada) conducted a series of 4 different surveys. The surveys included a face-to-face intercept survey at the Silver Bowl Park, a direct mail survey with local residents in the Silver Bowl Park area, a direct mail survey to local residents in the Desert Breeze Park vicinity, and face-to-face intercepts with attendees of the EcoJam Earth Day Event. A total of 883 persons participated in the survey (Alpha Communications Inc., 2001).

The majority (63.8 to 90.1 percent) of the responses were very positive, replying that the "...overall benefits of reclaimed water usage are very beneficial." There was a small minority who had concerns with "...environmental safety, bacteria, or germ build-up and general health risks to children" (Alpha Communications Inc., 2001). **Figure 7-3** shows a graphical representation of the average public opinion responses from the 4 surveys regarding reuse for 4 different uses: golf course irrigation, park irrigation, industrial cooling, and decorative water features.

Another portion of the survey asked if there were any benefits of using reclaimed water at park facilities. **Table 7-1** lists the responses.

There is no question that the public's enthusiasm for reuse (as noted in the cited studies) could reflect the hypothetical conditions set up by the survey questions and interviews used rather than signify a genuine willingness to endorse local funding of real programs that involve distribution of reclaimed water for nonpotable use in their neighborhood. Survey results do indicate, however, that, at least intellectually, "the public" is receptive to use of reclaimed water in well thought out programs. The results also support conclusions that this initial acceptance hinges in large measure on:

- The public's awareness of local water supply problems and perception of reclaimed water as having a place in the overall water supply allocation scheme
- Public understanding of the quality of reclaimed water and how it would be used
- Confidence in local management of the public utilities and in local application of modern technology
- Assurance that the reuse applications being considered involve minimal risk of accidental personal exposure

Table 7-1. Positive and Negative Responses to Potential Alternatives for Reclaimed Water

Use	Yes	No
Irrigation of Athletic Fields	84	16
Irrigation of Office Parks and Business Campuses	82	18
Irrigation of Highway Right-of-way	85	15
Residential Landscape Irrigation and Maintenance	74	26
Golf Course Irrigation	89	11
Irrigation of Agricultural Crops	82	18
Irrigation of Crops for Direct Human Consumption	30	70
Vehicle Wash Water	76	24
Concrete Production	90	10
Dust Control	82	18
Stream Augmentation	67	33
Toilet Flushing	80	20
Fire Protection	84	16
Ornamental Ponds/Fountains	56	44
Street Cleaning	87	13
Industrial Process Water	78	22
Wetland Creation	84	16
Pools/Spas	15	85
Potable Reuse – Direct	18	82
Potable Reuse – Indirect	40	60

Adapted from Hall and Rubin, 2002

7.4 Involving the Public in Reuse Planning

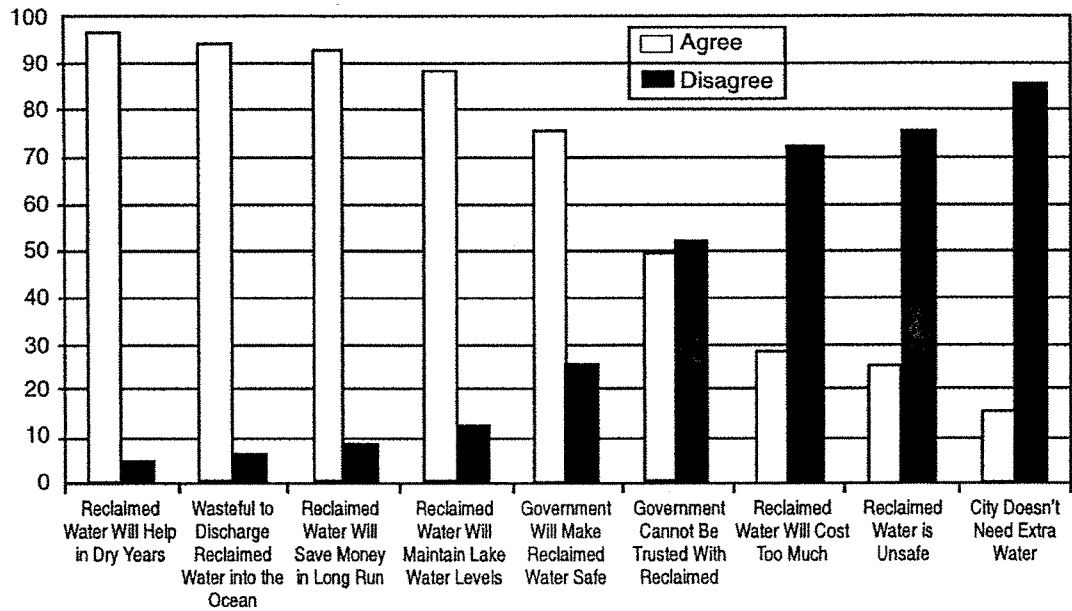
Even where water reclamation is common, there is a need to establish a flow of information to and from potential reuse customers, so that they can have a clear understanding of the program and provide input regarding their needs and concerns. Equally important is the need to address these concerns and answer any questions in a timely manner. This can help assure the public that their issues are being heard and that reuse planners are being forthcoming in their efforts.

Probably the most important step in encouraging the public acceptance is to establish and communicate the expected project benefits. If the project is intended to

extend water resources, then preliminary studies should address how much water will be made available through reclamation and compare the costs to those needed to develop other potable water sources. If reclamation costs are not competitive, then overriding non-economic issues must exist to equalize the value of the 2 sources. When reclamation is considered for environmental reasons, such as to reduce or eliminate surface water discharge, then the selected reuse alternative must also be competitive with other disposal options. Above all, the public must be aware of and understand all of the benefits.

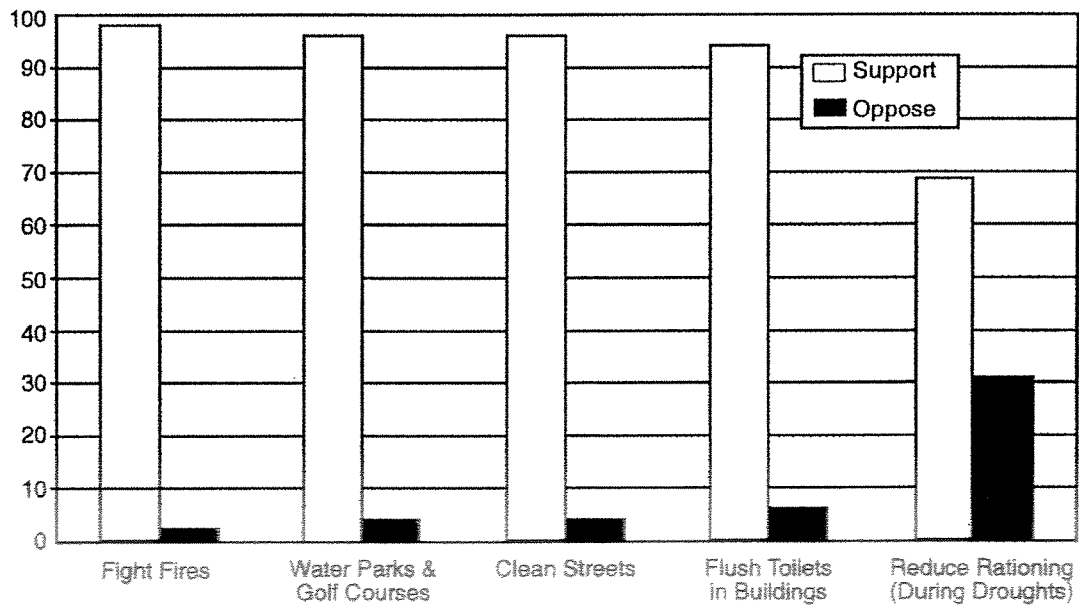
However, most potential reuse programs involve choices among systems with widely different economical and environmental impacts, which are of varying degrees of

Figure 7-1. Public Beliefs and Opinions



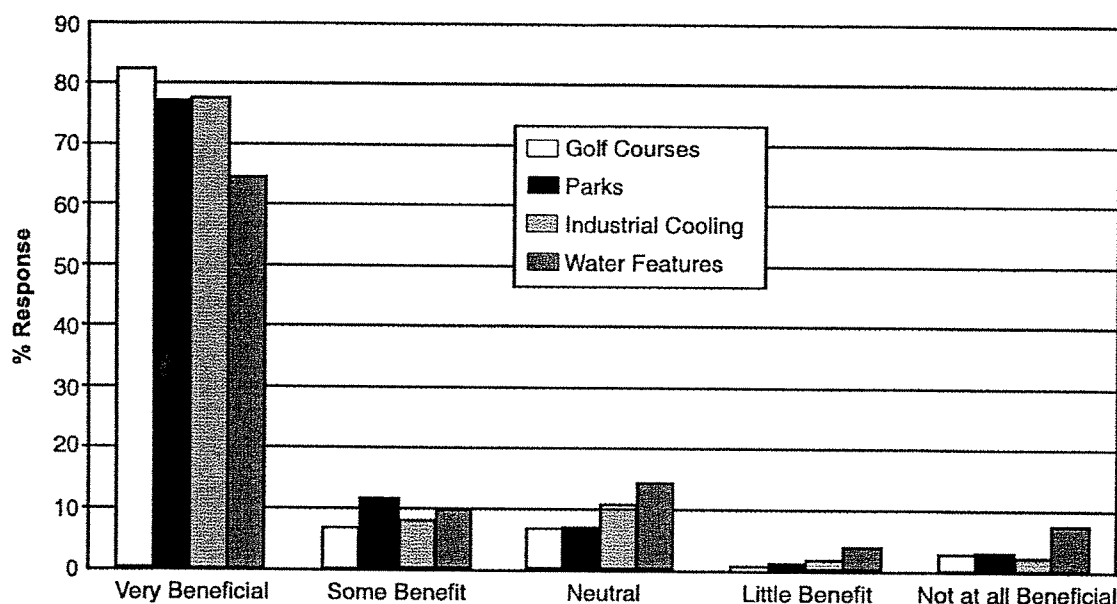
Adapted from Filice 1996

Figure 7-2. Support of Recycled Water Program Activities



Adapted from Filice 1996

Figure 7-3. Survey Results for Different Reuse



Data Source: Alpha Communications 2001

importance to many segments of the public. That is why development of the expected project benefits is so important because once they are firmly established, they become the plants of a public information program – the “why” the program is necessary and desirable. Without such validation, reclamation programs will be unable to withstand public scrutiny and the likelihood of project failure increases. In addition, only after the “why” is established can the “who” and “how” in public involvement truly be determined.

7.4.1 General Requirements for Public Participation

Figure 7-4 provides a flow chart of a public participation program for water reuse system planning.

The following items suggest an example approach that a community might consider in developing a reuse program. Note that information tools will vary depending upon how broad or involved an information program is needed.

- Determine, internally, the community’s reuse goals and the associated options and/or alternatives to be further considered.
- Identify any scientific/technical facts that exist, or are needed, to help explain the issues and alterna-

tives. If additional facts or studies are needed, consider beginning them in the earliest stages so that additional scientific data can be made available later in the process. Unanswered questions can damage the credibility of the program effort.

- Create a master list of stakeholders, including agencies, departments, elected officials, potential customers, and others who will be impacted in some way. It might be helpful to identify the level of interest different individuals and groups will have in the reuse planning process.
- Begin public outreach to specific target audiences in the form of informal meetings involving direct contact, limiting the number invited at any one time so that individual discussion is more easily accomplished
- Determine whether a task force or advisory committee is needed. If so, take steps to formally advertise and be sure to include representatives from the target audience groups. Plan a schedule and target date for reaching consensus on reuse alternatives; then plan well-prepared meetings that invite two-way communications. Bring in outside experts, such as scientists, to answer questions when needed.

Figure 7-4. Public Participation Program for Water Reuse System Planning

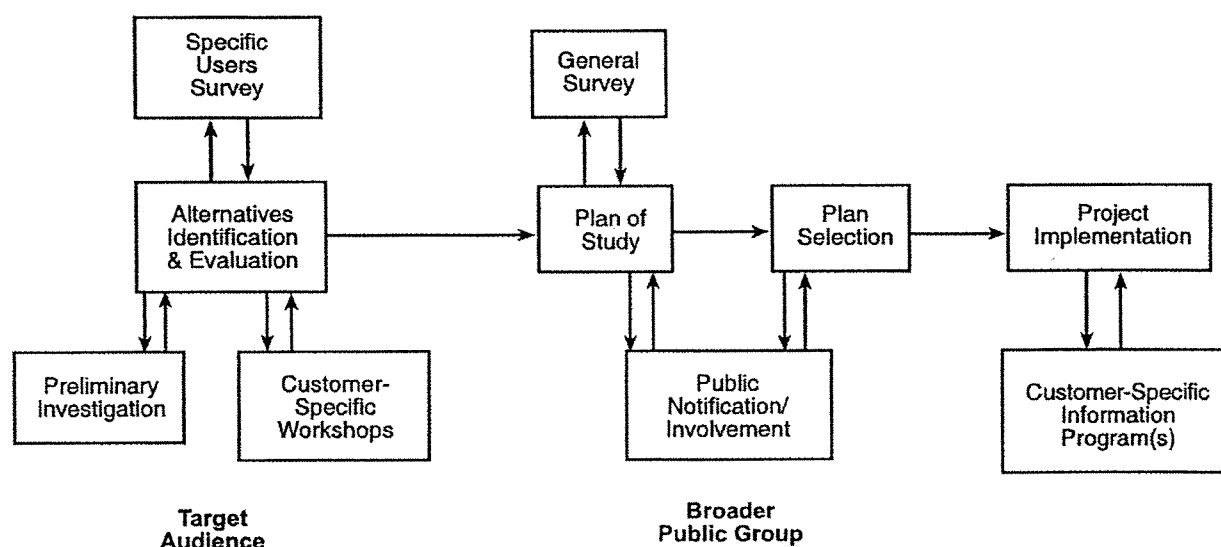


Table 7-2. Survey Results for Different Reuse

Purpose	Tools
Communitywide Education/Information	News media, editorial boards, program web site, traveling exhibits, brochures, educational videos, school programs, open houses
Direct Stakeholder or Citizen Contact	Neighborhood meetings, speeches and presentations to citizen/stakeholder groups, direct mail letters and surveys, program "hotlines" for answering information or managing construction complaints
Formalized Process	Public workshops, public meetings, presentations to elected bodies, public hearings, advisory committees, special task forces

From the task force or advisory committee, the community should be able to identify public issues that need further attention, and determine which additional public information tools will be needed. **Table 7-2** outlines a number of public information tools that can be used in the public participation process.

Once the issues are identified and public reaction is anticipated, the following tools may be useful in conveying information to the broader public:

- Citizen survey. Can be conducted via direct mail or telephone and might be accompanied by media releases to help increase the number of surveys returned or calls answered. In the early stages, a gen-

eral distribution survey may be helpful in identifying level of interest, potential customers, and any initial concerns that the population might have. Where specific concerns are identified, later public information efforts can be tailored to address them. These tailored efforts could include participation by other public agencies that can provide information on water reuse and regulatory requirements, informal discussions with some potential users to determine interest or fill data gaps, and initial background reports to appropriate local decision-making bodies.

- As the program progresses to alternative identification and evaluation, another survey might be considered. This survey could help confirm earlier re-

sults, monitor the effectiveness of the ongoing education program, or target specific users. Note that the percentage of citizens who take the time to participate in a survey varies widely from one community to another. This should not be the only tool relied upon in gathering input.

- **Open houses.** Advertise periodic public open houses where information is made available and knowledgeable people are on hand to answer questions. Maps, displays, and brief slide demonstrations are all useful open house tools.
- **Program website.** Increasingly, citizens are turning to websites as important information sources. Such a website can be purely informational or it can invite citizens to ask questions. The website should be updated on a regular basis and can include: its own survey or results of a citizen survey, answers to frequently asked questions, information regarding other successful programs in nearby communities, or a slideshow-style presentation that outlines the program goals and alternatives being considered.
- **Media relations.** In addition to project news releases, it can be very helpful to spend extra time with reporters who will be covering the topic on a regular basis, providing added background data, plant tours, and informal updates at appropriate times. This helps to provide accurate, balanced reports. The media can also be helpful in making survey data known, and in posting maps of construction areas once program implementation is underway.
- **Direct mail updates or occasional newspaper inserts.** These updates allow the community to address questions or issues - not relying specifically on a media report.
- **Briefings for government officials.** Because water reclamation programs often end up with a vote by a city council, county commission, or other elected body, it is vital that each elected official be well-informed throughout the reuse planning process. Therefore, informal briefings for individual officials can be an invaluable tool. These briefings are often conducted prior to public workshops and formal votes, and allow questions to be answered in advance of a larger, public setting.
- **Plant or project tours.** During the education process, a tour of an existing project that is similar to the one proposed can be an especially useful tool in providing information to key stakeholders, such as an advisory committee, elected body, or the media.

Once a reuse program has been determined, additional public information efforts will be needed throughout the implementation phase, including notification to citizens prior to construction occurring near their home or business. Then, as the reuse program goes on-line, additional media relations and direct mailings will be needed. In the case of urban reuse, this will include information to help homeowners through the connection process.

The City of Tampa's residential reclaimed water project (Florida) is one example of a successful comprehensive public participation program. The City used the services of Roberts Communication to conduct a targeted public education program, which included the following elements (Grosh *et al.*, 2002):

- Opinion leader interviews
- Public opinion survey
- Speakers bureau
- Direct mail to potential customers
- Newsletter article for homeowner association newsletters

7.4.1.1 Public Advisory Groups or Task Forces

If the scope or potential scope of the reuse program warrants (e.g., reclaimed water may be distributed to several users or types of users, or for a more controversial use), a public advisory group or task force can be formed to assist in defining system features and resolving problem areas. In its regulations for full-scale public participation programs, EPA requires that such group membership contain "substantially equivalent" representation from the private (non-interested), organized, representative, and affected segments of the public. It is recommended that, for reuse planning, group membership provide representation from potential users and their employees, interest groups, neighborhood residents, other public agencies, and citizens with specialized expertise in areas (such as public health) that pertain directly to reclamation/reuse.

The advantage of an advisory group or task force is that it offers an opportunity to truly educate a core group that may later become unofficial "spokespersons" for the project. For such a group to be successful, members must see that their input is being put to meaningful use. Depending upon the community need, either an advisory committee or task force may be appropriate. Advisory committees are generally formed for an indeterminate period to continuously provide input regard-

Table A-10. Indirect Potable Reuse

	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
State California	<ul style="list-style-type: none"> • Determined on a case-by-case basis • Based on all relevant aspects of each project, including the following factors: <ul style="list-style-type: none"> • treatment provided; • effluent quality and quantity; • spreading area operations; soil characteristics; • hydrogeology; • residence time and distance to withdrawal 	<ul style="list-style-type: none"> • Continuous on-line monitoring for turbidity before application of the disinfectant • Continuous monitoring for chlorine residual or for residual concentrations of other disinfectants • Treatment facilities designed to 	<ul style="list-style-type: none"> • Class I reliability - requires multiple or backup treatment units and a secondary power source • For treatment facilities required to provide full treatment and disinfection - minimum reject storage 	<ul style="list-style-type: none"> • System storage not required • If system storage is provided, at a minimum, system capacity shall be the volume equal to 3 times the portion of the average daily flow for which no alternative reuse or 	<ul style="list-style-type: none"> • Reasonable assurances must be provided that the hydraulic loading rates used in the design must enable the system to comply with the requirements while meeting applicable surface water and 	<ul style="list-style-type: none"> • Required • 1 upgradient well located as close as possible to the site without being affected by the site's discharge (background well) • 1 well at the edge of the zone of discharge down-gradient of the site 	<ul style="list-style-type: none"> • Outfalls for surface water discharges not to be located within 500 feet of existing or approved potable water intakes within Class I surface waters • Zones of discharge not to extend closer than 500 feet to a potable water 	<ul style="list-style-type: none"> • Involves the planned use of reclaimed water to augment Class F-1, G-1, or G-II groundwaters identified for potable water use and defined as groundwater recharge in regulations • Types of groundwater
Florida	<ul style="list-style-type: none"> • Discharge to Class I surface waters and to water contiguous to or tributary to Class I waters (less than 4 hours travel time): <ul style="list-style-type: none"> • Secondary treatment with filtration and high-level disinfection • Chemical feed facilities to be provided • 5 mg/l TSS 	<ul style="list-style-type: none"> • Continuous on-line monitoring for turbidity before application of the disinfectant • Continuous monitoring for chlorine residual or for residual concentrations of other disinfectants • Treatment facilities designed to 	<ul style="list-style-type: none"> • Class I reliability - requires multiple or backup treatment units and a secondary power source • For treatment facilities required to provide full treatment and disinfection - minimum reject storage 	<ul style="list-style-type: none"> • System storage not required • If system storage is provided, at a minimum, system capacity shall be the volume equal to 3 times the portion of the average daily flow for which no alternative reuse or 	<ul style="list-style-type: none"> • Reasonable assurances must be provided that the hydraulic loading rates used in the design must enable the system to comply with the requirements while meeting applicable surface water and 	<ul style="list-style-type: none"> • Required • 1 upgradient well located as close as possible to the site without being affected by the site's discharge (background well) • 1 well at the edge of the zone of discharge down-gradient of the site 	<ul style="list-style-type: none"> • Outfalls for surface water discharges not to be located within 500 feet of existing or approved potable water intakes within Class I surface waters • Zones of discharge not to extend closer than 500 feet to a potable water 	<ul style="list-style-type: none"> • Involves the planned use of reclaimed water to augment Class F-1, G-1, or G-II groundwaters identified for potable water use and defined as groundwater recharge in regulations • Types of groundwater

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<p>(single sample) to be achieved prior to disinfection</p> <ul style="list-style-type: none"> Total nitrogen - 10 mg/l (maximum annual average) Primary (except asbestos) and secondary drinking water standards must be met pH to fall within range established in secondary drinking water standards TOC <ul style="list-style-type: none"> - 3 mg/l (monthly average) - 5 mg/l (single sample) <p>Use of rapid-rate land application systems for projects considered reuse for groundwater recharge under 62-610.525:</p> <ul style="list-style-type: none"> Secondary treatment with filtration and 	<p>meet the full treatment and disinfection requirements to sample for TOC and total organic halogen daily, 7 days per week</p> <ul style="list-style-type: none"> Total coliforms and TSS analyzed daily if treatment facility is required to meet bacteriological requirements of the drinking water standards Parameters listed as primary drinking water standards that are imposed as reclaimed water limits to be analyzed monthly Parameters listed as secondary drinking water standards that are imposed 	<p>capacity equal to 3 day's flow at the average daily permitted flow of the treatment plant or the average daily permitted flow of the reuse system, whichever is less</p> <ul style="list-style-type: none"> If full treatment and disinfection is not required, the capacity requirement for reject storage shall be reduced to one day's flow Reject storage will not be required if another permitted reuse system or effluent disposal system is capable of discharging the reject water in accordance with requirements 	<p>disposal system is permitted</p> <ul style="list-style-type: none"> Water balance required with volume of storage based on a 10-year recurrence interval and a minimum of 20 years of climatic data Not required if alternative system is incorporated into the system design to ensure continuous facility operation 	<p>groundwater quality standards</p> <ul style="list-style-type: none"> A groundwater mounding analysis is to be included in the engineering report for projects involving discharges to groundwater and should provide reasonable assurances that the proposed project will function as intended and will not result in excessive mounding of groundwaters, increases in surface water elevations, property damage or interference with reasonable use of property within the affected area 	<p>(compliance well)</p> <ul style="list-style-type: none"> 1 well downgradient from the site and within the zone of discharge (intermediate well) 1 well located adjacent to unlined storage ponds or lakes Other wells may be required depending on site-specific criteria Quarterly monitoring required for water level, nitrate, total dissolved solids, arsenic, cadmium, chloride, chromium, lead, fecal coliform, pH, and sulfate Monitoring may be required for additional 	<p>supply well</p> <ul style="list-style-type: none"> 1,000 foot setback distance from injection well used for salinity barrier control to potable water supply wells Injection facilities: <ul style="list-style-type: none"> 500 feet to potable water supply wells that are existing or have been approved; Class I surface waters; or Class II Setback distance to Class I and Class II surface waters Setback distance to surface waters which are directly connected to Class F-1, G-1 or G-II groundwaters Indirect potable reuse involves the planned use of reclaimed water to 	<p>recharge systems include injection of reclaimed water into Class F-1, G-1, or G-II groundwaters, specific rapid-rate land application systems, use of reclaimed water to create barriers to the landward or upward migration of salt water within Class F-1, G-1, or G-II groundwaters and discharge to surface waters which are directly connected to Class F-1, G-1 or G-II groundwaters</p> <ul style="list-style-type: none"> Indirect potable reuse involves the planned use of reclaimed water to

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<p>high-level disinfection</p> <ul style="list-style-type: none"> Chemical feed facilities to be provided 5 mg/l TSS (single sample) to be achieved prior to disinfection Total nitrogen - 10 mg/l (maximum annual average) Primary (except asbestos and bacteriological parameters) and secondary drinking water standards must be met pH to fall within range established in secondary drinking water standards <p>Groundwater recharge by injection of Class G-1 and F-1 groundwaters and Class G-II groundwaters containing 3000 mg/l or less of</p>	<p>as reclaimed water limits to be analyzed quarterly</p> <ul style="list-style-type: none"> pH - daily Except for total coliforms and pH, 24-hour composite samples to be used for parameters listed as primary or secondary drinking water standards Unregulated organic contaminants to be sampled annually for some types of projects Monitoring for <i>Giardia</i> and <i>Cryptosporidium</i> required quarterly or one time during each 2-year period depending on type of project Parameters to be monitored and sampling frequency to 	<ul style="list-style-type: none"> Minimum system size of 0.1 mgd Staffing - 24 hrs/day, 7 days/wk for systems required to provide full treatment and disinfection reduced staffing requirement to 6 hrs/day, 7 days/wk may be approved for systems not required to provide full treatment with diversion of reclaimed water to reuse system only during periods of operator presence and other provisions for increased reliability 			<p>parameters based on site-specific conditions and groundwater quality</p>	<p>system or municipal operations</p> <ul style="list-style-type: none"> 100 feet to site property line Some setback distances may be reduced if certain treatment requirements are met and assurances are provided 	<p>augment surface water resources which are used or will be used for public water supplies and includes discharges to Class I surface waters and discharges to other surface waters which are directly or indirectly connected to Class I surface waters</p> <ul style="list-style-type: none"> Public notification and public hearing requirements in place for projects involving surface water discharges and underground injection Pilot testing is required for all projects that are required to provide full treatment and disinfection

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<p>TDS:</p> <ul style="list-style-type: none"> Same treatment and water quality requirements as discharge to Class I surface waters except additional requirement for total organic halogen must be met Total organic halogen (TOX) - 0.2 mg/l (monthly average) - 0.3 mg/l (single sample) Alternative TOC and TOX limitations may be approved if certain conditions are met <p>Groundwater recharge by injection of Class G-II groundwaters containing greater than 3000 mg/l of TDS:</p> <ul style="list-style-type: none"> Same treatment and water quality requirements 	<p>be identified in wastewater facility permit</p> <ul style="list-style-type: none"> Minimum schedule for sampling and testing based on system capacity 						

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<p>as discharge to Class I surface waters except TOC and secondary drinking water requirements do not apply</p> <ul style="list-style-type: none"> • Limitations to be met before injection to groundwater or discharge to surface waters 							
Hawaii	<ul style="list-style-type: none"> • Determined on a case-by-case basis • Reclaimed water used for groundwater recharge by surface or subsurface application shall be at all times of a quality that fully protects public health • Projects that are over an aquifer classified as potable, where the application rates exceed the consumptive 	<ul style="list-style-type: none"> • Determined on a case-by-case basis 	<ul style="list-style-type: none"> • Multiple or standby units required of sufficient capacity to enable effective operation with any one unit out of service • Alarm devices required for loss of power, high water levels, failure of pumps or blowers, high head loss on filters, high effluent turbidity, loss of coagulant or polymer feed, and loss of 	<ul style="list-style-type: none"> • 20 days storage required unless it can be demonstrated that another time period is adequate or that no storage is necessary • Storage requirements based on water balance using at least a 30-year record • Reject storage required with a volume equal to 1 day of flow at the average daily design flow 		<ul style="list-style-type: none"> • Required groundwater monitoring system may consist of a number of lysimeters and/or monitoring wells depending on site size, site characteristics, location, method of discharge, and other appropriate considerations • One well upgradient and two wells downgradient for project sites 		<ul style="list-style-type: none"> • Department of Health evaluation of proposed groundwater recharge projects and expansion of existing projects made on an individual case basis where the use of recycled water involves a potential risk to public health • Evaluation based on all relevant aspects of each project including

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<ul style="list-style-type: none"> evapotranspiration of the vegetative cover, will be designated as a recharge project 		<ul style="list-style-type: none"> chlorine residual Standby power source required for treatment plant and distribution pump stations 	<ul style="list-style-type: none"> Emergency system storage not required where an alternate effluent disposal system has been approved 		<ul style="list-style-type: none"> 500 acres or more One well within the wetted field area for each project whose surface area is greater than or equal to 1,500 acres One lysimeter per 200 acres One lysimeter for project sites that have greater than 40 but less than 200 acres Additional lysimeters may be necessary to address concerns of public health or environmental protection as related to variable characteristics of the subsurface or of the operations of the project 		<ul style="list-style-type: none"> treatment provided, effluent quality and quantity, effluent or application spreading area operation, soil characteristics, hydrogeology, residence time, and distance to withdrawal A public hearing or a public referendum is required for the DOH to review a request to augment a potable water supply by recharging the potable water supply aquifer with recycled water
Massachusetts	<ul style="list-style-type: none"> Secondary Filtration (possibly) 	<ul style="list-style-type: none"> pH - weekly or daily BOD - weekly 	<ul style="list-style-type: none"> EPA Class I Reliability standards may 	<ul style="list-style-type: none"> Immediate, permitted discharge 		<p>A groundwater monitoring plan is required and</p>	<ul style="list-style-type: none"> No wastewater discharges will be permitted in 	<ul style="list-style-type: none"> Refers to discharges into aquifer

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<ul style="list-style-type: none"> Disinfection pH 6 - 9 BOD - less than 10 mg/l or 30 mg/l Turbidity - less than 2 NTU or 5 NTU Fecal coliform - median of no detectable colonies/100 ml over continuous, running 7-day sampling periods, not to exceed 14/100 ml or 200/100 ml TSS - 5 mg/l or 10 mg/l Total nitrogen - less than 10 mg/l Class I Groundwater Permit Standards (SDWA Drinking Water Standards) 	<ul style="list-style-type: none"> Turbidity - continuous Fecal coliform - daily or twice per week Metals - quarterly TSS - weekly or twice per week Nitrogen - once or twice per week MS-2 phage - quarterly Total culturable viruses - quarterly Variable testing requirements UV intensity or chlorine residual - daily 	<ul style="list-style-type: none"> Two independent and separate sources of power Unit redundancy Additional storage 	<ul style="list-style-type: none"> alternatives are required for emergency situations 		<ul style="list-style-type: none"> <i>must accomplish the following goals:</i> <ul style="list-style-type: none"> Evaluates upgradient (background) groundwater quality Evaluates the performance of land use components that are considered part of the treatment process Evaluates the overall impact of the project on local groundwater quality Acts as an early warning system between the discharge and sensitive receptors 	<ul style="list-style-type: none"> the Zone I of any public water supply well defined as the area encompassing a maximum 400-foot radius around the wellhead (assuming a greater than 100,000 gpd withdrawal rate) Discharging to Zone IIs, defined as the entire extent of the aquifer deposits which could fall within and upgradient from the production well's capture zone based on the predicted drawdown after 180-day drought conditions at the approved pumping rate, will be permitted in circumstances where it is 	<ul style="list-style-type: none"> recharge areas as defined by Zone II boundaries of community water systems and groundwater discharges that will recharge reservoirs or tributaries to reservoirs New treatment plants located in approved Zone IIs with less than a 2 year groundwater travel time to the public water supply well must treat to the more rigorous of the two standards described Existing treatment plants that can demonstrate 4 or 5 feet of separation and where the well has not shown any evidence of water quality

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
Washington	<ul style="list-style-type: none"> Oxidized, coagulated, filtered, reverse-osmosis treated and disinfected Total coliform - 1/100 ml (7-day median) - 5/100 ml (single sample) 5 mg/l BOD and TSS (7-day mean) Turbidity - 0.1 NTU (monthly mean) - 0.5 NTU (maximum) Total nitrogen - 10 mg/l as N (annual mean) TOC - 1.0 mg/l 	<ul style="list-style-type: none"> Point of compliance is the point of direct recharge of reclaimed water into the underground BOD - 24-hour composite samples collected at least daily TSS - 24 hour composite samples collected at least daily Total coliform - grab samples collected at least daily and at a time when wastewater 	<ul style="list-style-type: none"> Warning alarms independent of normal power supply Back-up power source Emergency storage: short-term, 1 day; long-term, 20 days Multiple treatment units or storage or disposal options Qualified personnel available or on call at all times the system is operating 	<ul style="list-style-type: none"> Storage required when no approved alternative disposal system exists Storage volume established by determining storage period required for duration of a 10-year storm, using a minimum of 20 years of climatic data At a minimum, system storage capacity should be the volume equal to 3 times that 		<ul style="list-style-type: none"> Will be required and based on reclaimed water quality and quantity, site specific soil and hydrogeologic characteristics and other considerations For direct recharge into potable groundwater aquifers, monitoring wells, at a minimum, shall be located at points 500 feet and 1,000 feet (plus or minus 	<ul style="list-style-type: none"> The minimum horizontal separation distance between the point of direct recharge and withdrawal as a source of drinking water supply shall be 2,000 feet 	<ul style="list-style-type: none"> Defined as direct recharge to potable groundwater aquifers Reclaimed water shall be retained underground for a minimum of 12 months prior to being withdrawn as a source of drinking water supply Project evaluation based on all relevant aspects of each project, including treatment and

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Table A-10. Indirect Potable Reuse

State	Reclaimed Water Quality and Treatment Requirements	Reclaimed Water Monitoring Requirements	Treatment Facility Reliability	Storage Requirements	Loading Rates	Groundwater Monitoring	Setback Distances	Other
	<p>(monthly mean)</p> <ul style="list-style-type: none"> Water quality criteria for primary contaminants (except nitrate), secondary contaminants, radionuclides and carcinogens listed in Table 1 in Chapter 173-200 WAC and any other maximum contaminant levels pursuant to Chapter 246-290 WAC must be met Minimum chlorine residual of 1 mg/l after a contact time of 30 minutes based on peak hourly flow A chlorine residual of at least 0.5 mg/l to be maintained in the reclaimed water during conveyance to the point of recharge 	<p>characteristics are most demanding on the treatment facilities and disinfection procedures</p> <ul style="list-style-type: none"> Continuous on-line monitoring of turbidity and chlorine residual TOC - 24-hour composite samples collected at least daily Primary contaminants (except total coliform organisms), secondary contaminants, radionuclides, and carcinogens - 24-hour composite samples collected at least quarterly Total nitrogen - grab or 24-hour composite samples 		<p>portion of the average daily flow for which no alternative reuse or disposal system is permitted</p>		<p>10 percent) along the groundwater flow path from the point of recharge to the nearest point of withdrawal of groundwater used as a source of drinking water supply</p> <ul style="list-style-type: none"> Groundwater shall be sampled for TOC and primary contaminants, secondary contaminants, radionuclides, and carcinogens listed in Table 1 in Chapter 173-200 WAC Samples from monitoring wells shall be collected at least quarterly 		<p>treatment reliability provided, reclaimed water quality and quantity, use or potential use of groundwater, operation and management of the recharge facilities, soil characteristics, hydrogeology, residence time of the reclaimed water in the underground prior to withdrawal and distance from the recharge area to nearest point of withdrawal</p> <ul style="list-style-type: none"> A pilot plant study shall be performed prior to implementation of direct recharge into a potable groundwater aquifer

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ACTON BOARD OF HEALTH

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Town of Acton
Comprehensive Water Resources Management Plan
Citizens Advisory Committee
Indirect Potable Reuse Working Group

Meeting #2
6/30/2005
Acton Town Hall, Room 126

Call to Order 730pm

- I. Introductions
- II. Minutes from 6/1/05
- III. Update on Reuse Activities
- IV. Discussion of the four major topics focusing on Emerging Contaminants
 - 1) Emerging contaminants – detection and removal
 - 2) The timing of the implementation of the project and coincidence with regulatory, treatment technology, and political timelines
 - 3) Source reduction efforts for water use and pollutant removal
 - 4) Centralized IPR versus Decentralized IPR
- V. Discuss future meeting dates and sites

Adjourn by 845pm



INDIRECT POTABLE REUSE WORKING GROUP

Acton Board of Health - Telephone (978) 264-9634

Meeting Minutes

6/1/2005 Meeting

Room 126

Acton Town Hall

Attendees: *Brent Reagor, Acton Health Department (BR)
 Jim Gagliard, Woodard and Curran (JG)
 *Greta Eckhardt, Acton Resident, AWD Land-Water Use Committee (GE)
 *Eric Hilfer, Acton Resident, ACES, CAC (EH)
 Mary Michelman, Acton Resident, ACES (MM)
 Peter Shanahan, Acton Resident (PS)
 *Art Gagne, Acton Resident, CAC (AG)
*IPR Working Group Member

**With Attachments

The meeting was called to order at 7:15pm

BR introduced the IPR group, the mission statement, and a short synopsis of what is expected of the group by the CAC. He explained that the need for a disposal site for highly treated wastewater treatment plant effluent is the driving factor in the formation of this group.

The members of the group and guests introduced themselves.

The group discussed why the concept of Indirect Potable Reuse is emerging in New England, based upon local and regional hydrologic losses, encouragement from EOEA through the Massachusetts Water Policy, and advocacy from organizations like the New England Water Environment Association.

BR explained the group was seated to discuss this issue as a stakeholder input group, and the group is expected to give a written report to the CAC at their October meeting with one of three answers, along with justification for the answer:

- 1) Yes, Acton should pursue this concept
- 2) No, Acton should not pursue this concept
- 3) This concept is promising for Acton but additional questions must be answered prior to implementation

BR introduced 2 other members, Joanne Bissetta and Pat Cumings, who could not attend the meeting.

GE asked about the current status of regulations regarding reuse in Massachusetts based upon what she had read in the 2004 EPA Reuse Guidelines sections sent to the group. BR explained that MA currently uses a set of "Reclaimed Water Use Standards" set forth in a DEP policy document, and that the state is currently seating a committee to write a set of water reuse regulations.

MM asked is drinking water standards or wastewater standards are applied to effluent discharges in reuse situations. BR explained that drinking water standards are applied in these cases as the DEP develops the permitted limits of various constituents of effluent.

MM and GE expressed concern regarding trace organic chemicals, pharmaceutical by-products, estrogen mimics in effluent. BR explained this is the major emerging issue and the Town is part of a nationwide surveillance study for these compounds being conducted by the Johns Hopkins School of Public Health and will be sampling at the wastewater treatment facility for a broad range of those compounds. PS explained that the USGS and other institutions have done surveillance studies in both surface waters and drinking water supplies and have found part-per trillion levels of some of these compounds in places like Atlanta and the lower Mississippi River basin.

PS explained that these emerging compounds exist currently in most areas of the country and we are just unaware because of the previous inability to analyze water specifically for these compounds.

MM asked about concentration of effluent on wellfields versus a broad distribution of onsite systems. BR, AG, and PS explained that onsite wastewater systems do not achieve levels of treatment anywhere near those of modern wastewater treatment facilities like Acton. GE brought up the inability of control over what people flush down the drain, BR mentioned that in a sewer system this can be somewhat controlled with dilution, and the ability to halt a discharge if harmful contaminants are found.

MM asked about local hydrologic loss within the Fort Pond Brook and Nashoba Brook associated with an IPR discharge at the High Street wellfields. PS explained that an IPR discharge with a shortened travel time from discharge point to well intake would actually benefit the local streams as the withdrawals will not have as great an impact. MM stated she would like to see this topic explored not only at Adams Street, but would rather see a distributed approach. The group continued to discuss the current status of Zone II discharges from both small package treatment facilities and onsite systems across Acton and the current impact of those systems on our wells.

BR stated that this group has also drawn much interest from both the public and private sectors and the group may have some observers or other participants from time to time.

AG and EH asked about the current treatment levels at the WWTF versus what they may have to be in order to achieve IPR. EH spoke about the Denver study mentioned in the EPA Reuse Guidelines on efficacy of treatment processes related to the removal of emerging contaminants. BR stated this is a major research issue now as a multitude of treatment technologies must be tested.

GE spoke about source reduction of contaminants and flow through conservation efforts and public education programs. The group shared favorable opinions on this subject and spoke about the research conducted into wastewater flows by the Health Department.

The group agreed that four major topic areas need to be discussed. In order of importance, they are:

- 1) Emerging contaminants – detection and removal
- 2) The timing of the implementation of the project and coincidence with regulatory, treatment technology, and political timelines
- 3) Source reduction efforts for water use and pollutant removal
- 4) Centralized IPR versus Decentralized IPR

The group agreed to meet approximately once every three weeks, with the coordination to come from BR. One of the next meetings will be held at the Acton WWTF.

BR thanked the members and guests for attending and stated the next meeting date will be sent out shortly.

The meeting adjourned at 845pm

Respectfully Submitted,

Brent L. Reagor

Pharmaceuticals, Hormones, and Other Organic Wastewater Contaminants in U.S. Streams, 1999–2000: A National Reconnaissance

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To provide the first nationwide reconnaissance of the occurrence of pharmaceuticals, hormones, and other organic wastewater contaminants (OWCs) in water resources, the U.S. Geological Survey used five newly developed analytical methods to measure concentrations of 95 OWCs in water samples from a network of 139 streams across 30 states during 1999 and 2000. The selection of sampling sites was biased toward streams susceptible to contamination (i.e. downstream of intense urbanization and livestock production). OWCs were prevalent during this study, being found in 80% of the streams sampled. The compounds detected represent a wide range of residential, industrial, and agricultural origins and uses with 82 of the 95 OWCs being found during this study. The most frequently detected compounds were coprostanol (fecal steroid), cholesterol (plant and animal steroid), *N,N*-diethyltoluamide (insect repellent), caffeine (stimulant), triclosan (antimicrobial disinfectant), tri(2-chloroethyl)phosphate (fire retardant), and 4-nonylphenol (nonionic detergent metabolite). Measured concentrations for this study were generally low and

rarely exceeded drinking-water guidelines, drinking-water health advisories, or aquatic-life criteria. Many compounds, however, do not have such guidelines established. The detection of multiple OWCs was common for this study, with a median of seven and as many as 38 OWCs being found in a given water sample. Little is known about the potential interactive effects (such as synergistic or antagonistic toxicity) that may occur from complex mixtures of OWCs in the environment. In addition, results of this study demonstrate the importance of obtaining data on metabolites to fully understand not only the fate and transport of OWCs in the hydrologic system but also their ultimate overall effect on human health and the environment.

Introduction

The continued exponential growth in human population has created a corresponding increase in the demand for the Earth's limited supply of freshwater. Thus, protecting the integrity of our water resources is one of the most essential environmental issues of the 21st century. Recent decades have brought increasing concerns for potential adverse human and ecological health effects resulting from the production, use, and disposal of numerous chemicals that offer improvements in industry, agriculture, medical treatment, and even common household conveniences (1). Research has shown that many such compounds can enter the environment, disperse, and persist to a greater extent than first anticipated. Some compounds, such as pesticides, are intentionally released in measured applications. Others, such as industrial byproducts, are released through regulated and unregulated industrial discharges to water and air resources. Household chemicals, pharmaceuticals, and other consumables as well as biogenic hormones are released directly to the environment after passing through wastewater treatment processes (via wastewater treatment plants, or domestic septic systems), which often are not designed to remove them from the effluent (2). Veterinary pharmaceuticals used in animal feeding operations may be released to the environment with animal wastes through overflow or leakage from storage structures or land application (3). As a result, there are a wide variety of transport pathways for many different chemicals to enter and persist in environmental waters.

Surprisingly, little is known about the extent of environmental occurrence, transport, and ultimate fate of many synthetic organic chemicals after their intended use, particularly hormonally active chemicals (4), personal care products, and pharmaceuticals that are designed to stimulate a physiological response in humans, plants, and animals (1, 5). One reason for this general lack of data is that, until recently, there have been few analytical methods capable of detecting these compounds at low concentrations which might be expected in the environment (6). Potential concerns from the environmental presence of these compounds include abnormal physiological processes and reproductive impairment (7–12), increased incidences of cancer (13), the development of antibiotic-resistant bacteria (14–17), and the potential increased toxicity of chemical mixtures (18). For many substances, the potential effects on humans and aquatic ecosystems are not clearly understood (1, 2, 19).

The primary objective of this study is to provide the first nationwide reconnaissance of the occurrence of a broad suite of 95 organic wastewater contaminants (OWCs) including

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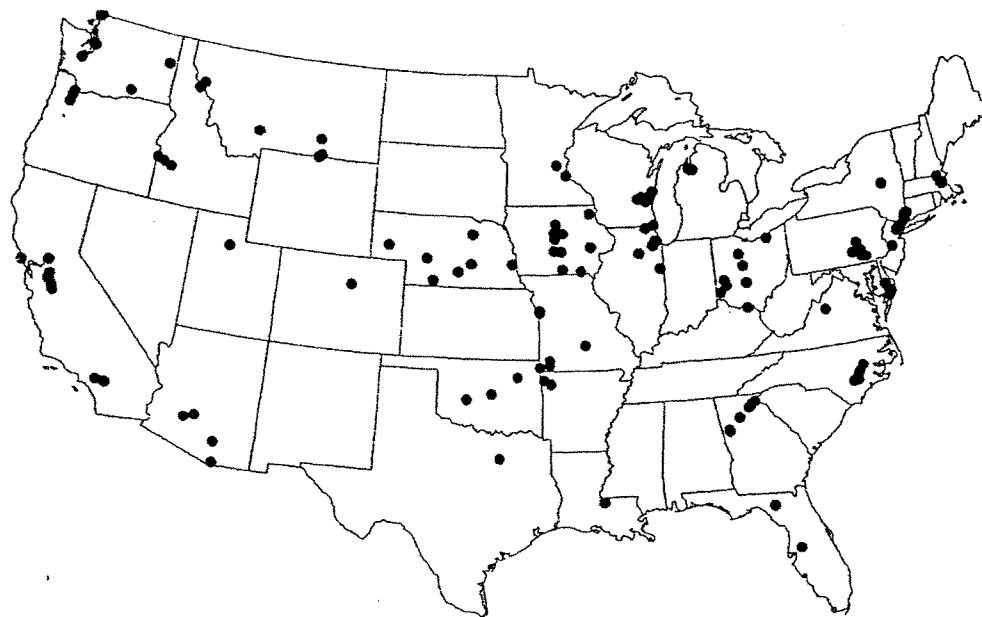


FIGURE 1. Location of 139 stream sampling sites.

many compounds of emerging environmental concern, in streams across the United States. These OWCs are potentially associated with human, industrial, and agricultural wastewaters and include antibiotics, other prescription drugs, nonprescription drugs, steroids, reproductive hormones, personal care products, products of oil use and combustion, and other extensively used chemicals. The target OWCs were selected because they are expected to enter the environment through common wastewater pathways, are used in significant quantities, may have human or environmental health implications, are representative or potential indicators of certain classes of compounds or sources, and/or can be accurately measured in environmental samples using available technologies. Although these 95 OWCs are just a small subset of compounds being used by society, they represent a starting point for this investigation examining the transport of OWCs to water resources of the United States.

This paper describes the analytical results available from 139 streams sampled during 1999–2000 (Figure 1). The results are intended to determine if OWCs are entering U.S. streams and to estimate the extent of their co-occurrence in susceptible waters. In addition, this study provides a focal point for the development and testing of new laboratory methods for measuring OWCs in environmental samples at trace levels, an interpretive context for future assessments of OWCs, and a means for establishing research priorities and future monitoring strategies. More complete interpretations, including an evaluation of the role of potential sources of contamination, will follow in subsequent papers.

Site Selection and Sampling

Little data were available on the occurrence of most of the targeted OWCs in U.S. streams at the onset of this investigation. Therefore, the selection of sampling sites primarily focused on areas considered susceptible to contamination from human, industrial, and agricultural wastewater. The 139 stream sites sampled during 1999–2000 (Figure 1) represent a wide range of geography, hydrogeology, land use, climate, and basin size. Specific information on the individual sampling sites is provided elsewhere (20).

All samples were collected by U.S. Geological Survey personnel using consistent protocols and procedures de-

signed to obtain a sample representative of the streamwaters using standard depth and width integrating techniques (21). At each site, a composite water sample was collected from about 4–6 vertical profiles which was split into appropriate containers for shipment to the participating laboratories. For those bottles requiring filtration, water was passed through a 0.7 μm , baked, glass-fiber filter in the field where possible, or else filtration was conducted in the laboratory. Water samples for each chemical analysis were stored in precleaned-amber, glass bottles and collected in duplicate. The duplicate samples were used for backup purposes (in case of breakage of the primary sample) and for laboratory replicates. Following collection, samples were immediately chilled and sent to the laboratory. To minimize contamination of samples, use of personal care items (i.e. insect repellents, colognes, perfumes), caffeinated products, and tobacco were discouraged during sample collection and processing.

Each stream site was sampled once during the 1999–2000 study period. Samples collected in 1999 were analyzed for a subset of the OWCs based on the watershed land-use characteristics. Samples collected in 2000 were analyzed for the complete suite of OWCs. The analytical results for each stream sample are available elsewhere (20).

Analytical Methods

To determine the environmental extent of 95 OWCs (Table 1) in susceptible streams, five separate analytical methods were used. Each method was developed independently in different laboratories, with somewhat different data objectives, such as identifying hormones versus identifying antibiotics. As a result of these differing objectives, varying approaches were used in the development of the five analytical methods. For example, select methods (Methods 1–3 below) used filtered water for solid-phase extraction (SPE) with liquid chromatography/mass spectrometry positive-ion electrospray (LC/MS-ESI(+)) analysis, while others (Methods 4 and 5 below) used whole-water continuous liquid–liquid extraction (CLLE) with capillary gas chromatography/mass spectrometry (GC/MS) analysis.

All methods use selected ion monitoring (SIM) for improved sensitivity; thus, only the target compounds were reported with no attempt to report data for nontarget.

TABLE 1. Summary of Analytical Results of Streams Sampled for 95 Organic Wastewater Contaminants¹

chemical (method)	CASRN	N	RL ($\mu\text{g/L}$)	freq (%)	max ($\mu\text{g/L}$)	med ($\mu\text{g/L}$)	use	MCL or HAL (2 σ) ($\mu\text{g/L}$)	lowest LC ₅₀ for the most sensitive indicator species ($\mu\text{g/L}$)/no. of aquatic studies identified (2 σ)
Veterinary and Human Antibiotics									
carbodox (1)	6804-07-5	104	0.10	0	ND	ND	antibiotic	—	—/1
chlortetracycline (1)	57-62-5	115	0.05	0	ND	ND	antibiotic	—	88000 ^a /3
chlortetracycline (2)	57-62-5	84	0.10	2.4	0.69	0.42	antibiotic	—	88000 ^a /3
ciprofloxacin (1)	85721-33-1	115	0.02	2.6	0.03	0.02	antibiotic	—	—/0
doxycycline (1)	564-25-0	115	0.1	0	ND	ND	antibiotic	—	—/0
enrofloxacin (1)	93106-60-6	115	0.02	0	ND	ND	antibiotic	—	40 ^a /29
erythromycin-H ₂ O (1)	114-07-8	104	0.05	21.5	1.7	0.1	erythromycin metabolite	—	665000 ^b /35
lincomycin (1)	154-21-2	104	0.05	19.2	0.73	0.06	antibiotic	—	—/0
norfloxacin (1)	70458-96-7	115	0.02	0.9	0.12	0.12	antibiotic	—	—/6
oxytetracycline (1)	79-57-2	115	0.1	0	ND	ND	antibiotic	—	102000 ^a /46
oxytetracycline (2)	79-57-2	84	0.10	1.2	0.34	0.34	antibiotic	—	102000 ^a /46
roxithromycin (1)	80214-83-1	104	0.03	4.8	0.18	0.05	antibiotic	—	—/0
sarafloxacin (1)	98105-99-8	115	0.02	0	ND	ND	antibiotic	—	—/0
sulfachloropyridazine (2)	80-32-0	84	0.05	0	ND	ND	antibiotic	—	—/0
sulfadimethoxine (1)	122-11-2	104	0.05	0	ND	ND	antibiotic	—	—/5
sulfadimethoxine (2)	122-11-2	84	0.05	1.2	0.06	0.06	antibiotic	—	—/5
sulfamerazine (1)	127-79-7	104	0.05	0	ND	ND	antibiotic	—	100000 ^a /17
sulfamerazine (2)	127-79-7	84	0.05	0	ND	ND	antibiotic	—	100000 ^a /17
sulfamethazine (1)	57-68-1	104	0.05	4.8	0.12	0.02	antibiotic	—	100000 ^a /17
sulfamethazine (2)	57-68-1	84	0.05	1.2	0.22	0.22	antibiotic	—	100000 ^a /17
sulfamethizole (1)	144-82-1	104	0.05	1.0	0.13	0.13	antibiotic	—	—/0
sulfamethoxazole (1)	723-46-6	104	0.05	12.5	1.9	0.15	antibiotic	—	—/0
sulfamethoxazole (3)	723-46-6	84	0.023	19.0	0.52	0.066	antibiotic	—	—/0
sulfathiazole (1)	72-14-0	104	0.10	0	ND	ND	antibiotic	—	—/0
sulfathiazole (2)	72-14-0	84	0.05	0	ND	ND	antibiotic	—	—/0
tetracycline (1)	60-54-8	115	0.05	0	ND	ND	antibiotic	—	550000 ^b /3
tetracycline (2)	60-54-8	84	0.10	1.2	0.11	0.11	antibiotic	—	550000 ^b /3
trimethoprim (1)	738-70-5	104	0.03	12.5	0.71	0.15	antibiotic	—	3000 ^a /4
trimethoprim (3)	738-70-5	84	0.014	27.4	0.30	0.013	antibiotic	—	3000 ^a /4
tylosin (1)	1401-69-0	104	0.05	13.5	0.28	0.04	antibiotic	—	—/0
virginiamycin (1)	21411-53-0	104	0.10	0	ND	ND	antibiotic	—	—/0
Prescription Drugs									
albuterol (salbutamol) (3)	18559-94-9	84	0.029	0	ND	ND	asthmatic	—	—/0
cimetidine (3)	51481-61-9	84	0.007	9.5	0.58 ^d	0.074 ^d	antacid	—	—/0
codeine (3)	76-57-3	46	0.24	6.5	0.019	0.012	analgesic	—	—/0
codeine (4)	76-57-3	85	0.1	10.6	1.0 ^d	0.2 ^d	analgesic	—	—/0
dehydronifedipine (3)	67035-22-7	84	0.01	14.3	0.03	0.012	antihypertensive	—	—/0
digoxin (3)	20830-75-5	46	0.26	0	ND ^d	ND ^d	cardiac stimulant	—	1000000 ^a /24
digoxigenin (3)	1672-46-4	84	0.008	0	ND	ND	digoxin metabolite	—	—/0
diltiazem (3)	42399-41-7	84	0.012	13.1	0.049	0.021	antihypertensive	—	—/0
enalaprilat (3)	76420-72-9	84	0.15	1.2	0.046 ^d	0.046 ^d	enalapril maleate (antihypertensive) metabolite	—	—/0
fluoxetine (3)	54910-89-3	84	0.018	1.2	0.012 ^d	0.012 ^d	antidepressant	—	—/0
gemfibrozil (3)	25812-30-0	84	0.015	3.6	0.79	0.048	antihyperlipidemic	—	—/0
metformin (3)	657-24-9	84	0.003	4.8	0.15 ^d	0.11 ^d	antidiabetic	—	—/0
paroxetine metabolite (3)	—	84	0.26	0	ND ^d	ND ^d	paroxetine (antidepressant) metabolite	—	—/0
ranitidine (3)	66357-35-5	84	0.01	1.2	0.01 ^d	0.01 ^d	antacid	—	—/0
warfarin (3)	81-81-2	84	0.001	0	ND	ND	anticoagulant	—	16000 ^a /33
Nonprescription Drugs									
acetaminophen (3)	103-90-2	84	0.009	23.8	10	0.11	antipyretic	—	6000 ^a /14
caffeine (3)	58-08-2	84	0.014	61.9	6.0	0.081	stimulant	—	40000 ^a /77
caffeine (4)	58-08-2	85	0.08	70.6	5.7	0.1	stimulant	—	40000 ^a /77
cotinine (3)	486-56-6	84	0.023	38.1	0.90	0.024	nicotine metabolite	—	—/0
cotinine (4)	486-56-6	54	0.04	31.5	0.57	0.05	nicotine metabolite	—	—/0
1,7-dimethylxanthine (3)	611-59-6	84	0.018	28.6	3.1 ^d	0.11 ^d	caffeine metabolite	—	—/0
ibuprofen (3)	15687-27-1	84	0.018	9.5	1.0	0.20	antiinflammatory	—	—/0
Other Wastewater-Related Compounds									
1,4-dichlorobenzene (4)	106-46-7	85	0.03	25.9	4.3	0.09	deodorizer	75	1100 ^a /190
2,6-di- <i>tert</i> -butylphenol (4)	128-39-2	85	0.08	3.5	0.11 ^d	0.06 ^d	antioxidant	—	—/2
2,6-di- <i>tert</i> -butyl-1,4-benzoquinone (4)	719-22-2	85	0.10	9.4	0.46	0.13	antioxidant	—	—/0
5-methyl-1H-benzotriazole (4)	136-85-6	54	0.10	31.5	2.4	0.39	anticorrosive	—	—/0
acetophenone (4)	98-86-2	85	0.15	9.4	0.41	0.15	fragrance	—	155000 ^a /21
anthracene (4)	120-12-7	85	0.05	4.7	0.11	0.07	PAH	—	54 ^a /186
benzo[a]pyrene (4)	50-32-6	85	0.05	9.4	0.24	0.04	PAH	0.2	1.5 ^a /428
3- <i>tert</i> -butyl-4-hydroxy anisole (4)	25073-16-5	85	0.12	2.4	0.2 ^c	0.1 ^c	antioxidant	—	870 ^a /14
butylated hydroxy toluene (4)	126-37-0	85	0.08	2.4	0.1 ^c	0.1 ^c	antioxidant	—	1440 ^a /15
bis(2-ethylhexyl) adipate (4)	103-23-1	85	2.6	3.6	10 ^a	3 ^a	plasticizer	400	480 ^a /5
bis(2-ethylhexyl) phthalate (4)	117-81-7	85	2.6	10.6	20 ^a	7 ^a	plasticizer	6	7500 ^a /305

TABLE 1. (Continued)

chemical (method)	CASRN	N	RL ($\mu\text{g/L}$)	freq (%)	max ($\mu\text{g/L}$)	med ($\mu\text{g/L}$)	use	MCL or HAL (23) ($\mu\text{g/L}$)	lowest LC ₅₀ for the most sensitive indicator species ($\mu\text{g/L}$)/no. of aquatic studies identified (24)
Other Wastewater-Related Compounds									
bisphenol A (4)	80-05-7	85	0.09	41.2	12	0.14	plasticizer	—	3600*/26
carbaryl (4)	63-25-2	85	0.06	16.5	0.1 ^a	0.04 ^a	insecticide	700	0.4*/1541
cis-chlordane (4)	5103-71-9	85	0.04	4.7	0.1	0.02	insecticide	2	7.4*/28
chlorpyrifos (4)	2921-88-2	85	0.02	15.3	0.31	0.06	insecticide	20	0.1*/1794
diazinon (4)	333-41-5	85	0.03	25.9	0.35	0.07	insecticide	0.6	0.56*/1040
dieldrin (4)	60-57-1	85	0.08	4.7	0.21	0.18	insecticide	0.2	2.6*/1540
diethylphthalate (4)	84-66-2	54	0.25	11.1	0.42	0.2	plasticizer	—	12000*/129
ethanol,2-butoxy-phosphate (4)	78-51-3	85	0.2	45.9	6.7	0.51	plasticizer	—	10400*/7
fluoranthene (4)	206-44-0	85	0.03	29.4	1.2	0.04	PAH	—	74*/216
lindane (4)	58-89-9	85	0.05	5.9	0.11	0.02	insecticide	0.2	30*/1979
methyl parathion (4)	298-00-0	85	0.06	1.2	0.01	0.01	insecticide	2	12*/888
4-methyl phenol (4)	106-44-5	85	0.04	24.7	0.54	0.05	disinfectant	—	1400*/74
naphthalene (4)	91-20-3	85	0.02	16.5	0.08	0.02	PAH	20	910*/519
N,N-diethyltoluamide (4)	134-62-3	54	0.04	74.1	1.1	0.06	insect repellent	—	71250*/9
4-nonylphenol (4)	251-545-23	85	0.50	50.6	40 ^g	0.8 ^g	nonionic detergent metabolite	—	130*/135
4-nonylphenol monoethoxylate (4)	—	85	1.0	45.9	20 ^g	1 ^g	nonionic detergent metabolite	—	14450*/4
4-nonylphenol diethoxylate (4)	—	85	1.1	36.5	9 ^g	1 ^g	nonionic detergent metabolite	—	5500*/6
4-octylphenol monoethoxylate (4)	—	85	0.1	43.5	2 ^g	0.2 ^g	nonionic detergent metabolite	—	—/0
4-octylphenol diethoxylate (4)	—	85	0.2	23.5	1 ^g	0.1 ^g	nonionic detergent metabolite	—	—/0
phenanthrene (4)	85-01-8	85	0.06	11.8	0.53	0.04	PAH	—	590*/192
phenol (4)	108-95-2	85	0.25	8.2	1.3 ⁱ	0.7 ⁱ	disinfectant	400	4000*/2085
phthalic anhydride (4)	85-44-9	85	0.25	17.6	1 ⁱ	0.7 ⁱ	plastic manufacturing	—	40400*/5
pyrene (4)	129-00-0	85	0.03	28.2	0.84	0.05	PAH	—	90.9*/112
tetrachloroethylene (4)	127-18-4	85	0.03	23.5	0.70 ^a	0.07 ^a	solvent, degreaser	5	4680*/147
triclosan (4)	3380-34-5	85	0.05	57.6	2.3	0.14	antimicrobial disinfectant	—	180*/3
tri(2-chloroethyl) phosphate (4)	115-96-8	85	0.04	57.6	0.54	0.1	fire retardant	—	66000*/8
tri(dichlorisopropyl) phosphate (4)	13674-87-8	85	0.1	12.9	0.16	0.1	fire retardant	—	3600*/9
triphenyl phosphate (4)	115-86-6	85	0.1	14.1	0.22	0.04	plasticizer	—	280*/66
Steroids and Hormones									
cis-androsterone (5)	53-41-8	70	0.005	14.3	0.214	0.017	urinary steroid	—	—/0
cholesterol (4)	57-88-5	85	1.5	55.3	10 ^d	1 ^d	plant/animal steroid	—	—/0
cholesterol (5)	57-88-5	70	0.005	84.3	60 ^b	0.83	plant/animal steroid	—	—/0
coprostanol (4)	360-68-9	85	0.6	35.3	9.8 ^d	0.70 ^d	fecal steroid	—	—/0
coprostanol (5)	360-68-9	70	0.005	85.7	150 ^b	0.088	fecal steroid	—	—/0
equilenin (5)	517-09-9	70	0.005	2.8	0.278	0.14	estrogen replacement	—	—/0
equilin (5)	474-86-2	70	0.005	1.4	0.147	0.147	estrogen replacement	—	—/0
17 α -ethynyl estradiol (5)	57-63-6	70	0.005	15.7	0.831	0.073	ovulation inhibitor	—	—/22
17 α -estradiol (5)	57-91-0	70	0.005	5.7	0.074	0.03	reproductive hormone	—	—/0
17 β -estradiol (4)	50-28-2	85	0.5	10.6	0.2 ^d	0.16 ^d	reproductive hormone	—	—/0
17 β -estradiol (5)	50-28-2	70	0.005	10.0	0.093	0.009	reproductive hormone	—	—/0
estriol (5)	50-27-1	70	0.005	21.4	0.051	0.019	reproductive hormone	—	—/0
estrone (5)	53-16-7	70	0.005	7.1	0.112	0.027	reproductive hormone	—	—/11
mestranol (5)	72-33-3	70	0.005	10.0	0.407	0.074	ovulation inhibitor	—	—/0
19-norethisterone (5)	68-22-4	70	0.005	12.8	0.872	0.048	ovulation inhibitor	—	—/0
progesterone (5)	57-83-0	70	0.005	4.3	0.199	0.11	reproductive hormone	—	—/0
stigmastanol (4)	19466-47-8	54	2.0	5.6	4 ^d	2 ^d	plant steroid	—	—/0
testosterone (5)	58-22-0	70	0.005	2.8	0.214	0.116	reproductive hormone	—	—/4

* *Daphnia magna* (water flea) — 48 h exposure LC₅₀. ^a Other species and variable conditions. ^c *Oncorhynchus mykiss* (rainbow trout) — 96 h exposure LC₅₀. ^d Concentration estimated — average recovery <60%. ^e *Pimephales promelas* (fathead minnow) — 96 h exposure LC₅₀. ^f Concentration estimated — compound routinely detected in laboratory blanks. ^g Concentration estimated — reference standard prepared from a technical mixture. ^h Concentration estimated — value greater than highest point on calibration curve. ⁱ Compounds suspected of being hormonally active are in bold (4, 22). CASRN, Chemical Abstracts Service Registry Number; N, number of samples; RL, reporting level; freq, frequency of detection; max, maximum concentration; med, median detectable concentration; MCL, maximum contaminant level; HAL, health advisory level; LC₅₀, lethal concentration with 50% mortality; ND, not detected; —, not available; PAH, polycyclic aromatic hydrocarbon.

compounds. Target compounds within each method were selected from the large number of chemical possibilities based upon usage, toxicity, potential hormonal activity, and persistence in the environment. Some compounds that fit the above criteria, however, could not be included (such as amoxicillin, roxarsone, polybrominated diphenyl ethers) because they were either incompatible with the corresponding method or reference standards were not available. Positive identification of a compound required elution within the expected retention time window. In addition, the sample

spectra and ion abundance ratios were required to match that of the reference standard compounds. The base-peak ion was used for quantitation, and, if possible, two qualifier ions were used for confirmation. After qualitative criteria were met, compound concentrations were calculated from 5 to 8 point calibration curves (generally from 0.01 to 10.0 $\mu\text{g/L}$) using internal standard quantitation. Methods 1 and 2 process calibration standards through the extraction procedure which generally corrects concentrations for method losses but not matrix effects. Methods 3–5 do not

extract calibration standards, thus the reported concentrations are not corrected for method losses. Reporting levels (RLs) were determined for each method by either an evaluation of instrument response, calculation of limit of detection, or from a previously published procedure (25). RLs were adjusted based on experience with the compounds in each method, known interferences, or known recovery problems.

The following descriptions are intended to provide a brief overview of the five analytical methods used for this study. More comprehensive method descriptions are provided elsewhere (26–28) or will be available in subsequent publications.

Method 1. This method targets 21 antibiotic compounds (Table 1) in 500-mL filtered water samples using modifications from previously described methods (26, 29). The antibiotics were extracted and analyzed by tandem SPE and single quadrupole, LC/MS-ESI(+) using SIM. To prevent the tetracycline antibiotics from complexing with Ca^{2+} and Mg^{2+} ions and residual metals on the SPE cartridges, 0.5 mg of disodium ethylenediaminetetraacetate (Na_2EDTA ; $\text{C}_{10}\text{H}_{14}\text{O}_8\text{Na}_2\text{N}_2\text{H}_2\text{O}$) was added to each water sample. Sample pH was adjusted to 3 using concentrated H_2SO_4 . The tandem SPE included an Oasis Hydrophilic-Lipophilic-Balance (HLB) cartridge (60 mg) followed by a mixed mode, HLB-cation exchange (MCX) cartridge (60 mg) (Waters Inc., Milford, MA). The HLB and MCX cartridges were conditioned with ultrapure H_2O , CH_3OH , and CH_3OH with 5% NH_4OH . The HLB cartridge was attached to the top of the MCX cartridge, and the sample was passed through the SPE cartridges using a vacuum extraction manifold. The cartridges were eluted with CH_3OH , and the MCX cartridge was eluted separately using CH_3OH with 5% NH_4OH . The eluate was spiked with 500 ng of $^{13}\text{C}_6$ -sulfamethazine (internal standard), vortexed, and evaporated to 20 μL using N_2 and a water bath of 55°C. Three hundred μL of 20 mM of $\text{NH}_4\text{C}_2\text{H}_3\text{OO}$ (pH 5.7) was added to sample eluate, vortexed, transferred to a glass chromatography vial, and frozen until analysis. Samples were extracted as a set of 11 environmental samples, one duplicate sample, two fortified ultrapure water spikes (check standards), and two ultrapure water blanks.

Method 2. This method targets eight antibiotic compounds (Table 1) in filtered water samples. Complete details of this method have been described previously (26). The antibiotics were extracted and analyzed using SPE and SIM LC/MS-ESI(+). Samples were prepared for extraction by adding $^{13}\text{C}_6$ -sulfamethazine and meclocycline as surrogate standards, Na_2EDTA , and H_2SO_4 . Target compounds were extracted using 60-mg HLB cartridges preconditioned with CH_3OH , NHCl , and distilled H_2O . Target compounds were eluted with CH_3OH into a test tube containing the internal standard, simatone. The extracts were then concentrated under N_2 to approximately 50 μL , and mobile phase A (10 mM $\text{NH}_4\text{H}_2\text{O}_2$ in 90/10 water/ CH_3OH with 0.3% CH_2O_2) was added. The resulting solutions were transferred to amber autosampler vials to prevent photodegradation of tetracyclines (30). Mobile phase conditions are described in detail elsewhere (26).

For each compound, the proton adduct of the molecular ion ($\text{M} + \text{H}$)⁺ and at least one confirming ion were acquired using LC/MS-ESI(+). All mass spectral conditions are described in detail elsewhere (26). Quantitation was based on the ratio of the base peak ion ($\text{M} + \text{H}$)⁺ of the analyte to the base peak of the internal standard. Standard addition was used for quantitation where each sample was analyzed with and without the addition of a 0.5 $\mu\text{g/L}$ spike to correct for suppression of the electrospray signal.

Method 3. This method targets 21 human prescription and nonprescription drugs and their select metabolites (Table 1) in filtered water samples. Compounds were extracted from

1 L water samples using SPE cartridges that contain 0.5 g of HLB (flow rate of 15 mL/min). After extraction, the adsorbed compounds were eluted with CH_3OH followed by CH_3OH acidified with $\text{C}_2\text{HCl}_3\text{O}_2$. The two fractions were reduced under N_2 to near dryness and then combined and brought to a final volume of 1 mL in 10% $\text{C}_2\text{H}_3\text{N}$:90% H_2O buffered with $\text{NH}_4\text{H}_2\text{O}_2/\text{CH}_2\text{O}_2$.

Compounds were separated and measured by high-performance liquid chromatography (HPLC) using a polar (neutral silanol) reverse-phase octylsilane (C8) HPLC column (Metasil Basic 3 μm , 150 \times 2.0 mm; Metachem Technologies). The compounds were eluted with a binary gradient of mobile phase A (aqueous $\text{NH}_4\text{H}_2\text{O}_2/\text{CH}_2\text{O}_2$ buffer; 10 mM, pH 3.7) and mobile phase B (100% $\text{C}_2\text{H}_3\text{N}$).

Method 4. This method (27, 28) targets 46 OWCs (Table 1) in unfiltered water. One-liter whole-water samples were extracted using CLLE with CH_2Cl_2 . Distilled solvent was recycled through a microdroplet dispersing frit to improve extraction efficiency. Samples were extracted for 3 h at ambient pH and for an additional 3 h at pH 2. The extract was concentrated under N_2 to 1 mL and analyzed by capillary-column GC/MS. Available standards for the 4-nonylphenol compounds were composed of multiple isomers, and thus, laboratory standards for these compounds as well as octylphenol ethoxylates were prepared from technical mixtures.

Method 5. This method (28) targets 14 steroid compounds including several biogenic and synthetic reproductive hormones (Table 1). The CLLE extracts from the previously analyzed samples of Method 4 were derivatized and reanalyzed. Analysis of steroid and hormone compounds by GC/MS is enhanced by derivatization to deactivate the hydroxyl and keto functional groups. The technique used in this study is the formation of trimethylsilyl (TMS) ethers of the hydroxyl groups and oximes of the keto groups. Samples were stored in a silylating reagent to prevent hydrolysis of the derivatives back to the free compound. Surrogate standards (d_4 estradiol and d_7 cholesterol) were added to the samples prior to derivatization to evaluate method performance. After derivatization, the samples were analyzed by GC/MS.

Quality Assurance Protocol. At least one fortified laboratory spike and one laboratory blank was analyzed with each set of 10–16 environmental samples. Most methods had surrogate compounds added to samples prior to extraction to monitor method performance. A summary of recoveries for target compounds and surrogate compounds in environmental samples (Table 2) indicates the general proficiency of the methods. The RL (Table 1) is equivalent to the lowest concentration standard that could be reliably quantitated. The compound concentrations reported below the RL or the lowest calibration standard were estimated as indicated in Figure 2. The concentration of compounds with <60% recovery, routinely detected in laboratory blanks, or prepared with technical grade mixtures, was also considered estimated (Table 1).

The laboratory blanks were used to assess potential sample contamination. Blank contamination was not subtracted from environmental results. However, environmental concentrations within twice the values observed in the set blank were reported as less than the RL.

A field quality assurance protocol was used to determine the effect, if any, of field equipment and procedures on the concentrations of OWCs in water samples. Field blanks, made from laboratory-grade organic free water, were submitted for about 5% of the sites and analyzed for all of the 95 OWCs. Field blanks were subject to the same sample processing, handling, and equipment as the stream samples. To date, one field blank had a detection of coprostanol and testosterone, one field blank had a detection of naphthalene and tris(1-chloroisopropyl)phosphate, and one field blank had

TABLE 2. Summary of Quality Assurance/Quality Control Results for Target and Surrogate Compounds^a

compound	spike concn (µg/L)	mean % recovery	% RSD
Method 1			
target compounds	1.0	99.0	12.1
Method 2			
target compounds	1.0	97.5	12.2
¹³ C ₆ -sulfamethazine	1.0	80.0	20.0
meclocycline	1.0	80.0	20.0
Method 3			
target compounds	0.5	85.1	11.6
C ₁₃ -phenacetin	1.0	96.8	14.0
Method 4			
target compounds	1.0	81.0	11.0
d ₂₁ -BHT	2.0	63.0	25.0
<i>n</i> -nonylphenol	2.0	83.0	20.0
Method 5			
target compounds	NA	NA	NA
d ₄ -estradiol ^a	0.047	128.8	42.0
d ₃ -testosterone ^a	0.051	148.5	47.3
d ₇ -cholesterol ^a	0.053	116.9	55.9

^a Surrogate standard added after CCLE extraction but prior to derivatization. ^b RSD, relative standard deviation; NA, not currently available.

a detection of naphthalene, 4-nonylphenol, phenol, 4-*tert*-octylphenol monoethoxylate, and ethanol,2-butoxy-phosphate. Most of these detections were near their respective

RLs verifying the general effectiveness of the sampling protocols used for this study. In addition all field blanks had low level concentrations of cholesterol being measured using Method 5 (median concentration = 0.09 µg/L) documenting its ubiquitous nature in the environment. Cholesterol concentrations from 0.005 to 0.18 µg/L obtained through Method 5 were set to less than the RL.

Compounds that were measured by more than one analytical method (Table 1; Figure 3) also were used to evaluate the results for this study. The presence or absence of these compounds were confirmed in 100% of the determinations for sulfamerazine, and sulfathiazole; 98.8% for oxytetracycline, sulfadimethoxine, sulfamethazine, and tetracycline; 98.6% for cholesterol and coprostanol; 97.6% for chlortetracycline; 95.7% for 17β-estradiol; 94.4% for cotinine; 94.0% for trimethoprim; 89.1% for sulfamethoxazole; 86.4% for codeine; and 83.3% for caffeine. The comparisons for codeine, caffeine, and cotinine may have been affected by the differing extractions (SPE versus CLLE) as well as differing types of sample (filtered versus whole water).

An interlaboratory comparison of Methods 1 and 3 was conducted using two reagent water blanks and 24 reagent water spikes prepared at concentrations ranging from 0.5 to 1.1 µg/L for two frequently detected antibiotics (sulfamethoxazole and trimethoprim). The results demonstrated that both methods are accurately confirming the presence of sulfamethoxazole and trimethoprim in water, with the measured concentrations being within a factor of 3 or better of the actual concentrations for these compounds. No false positives or false negatives occurred for this experiment.

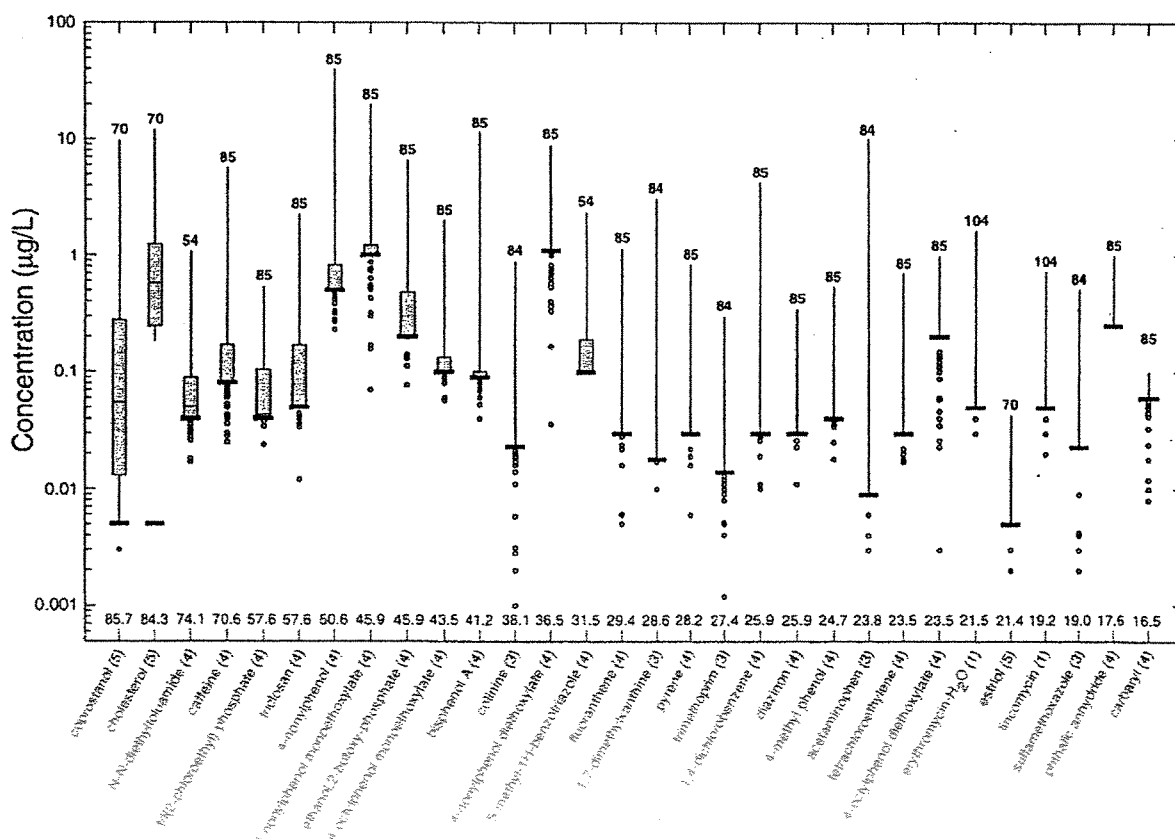


FIGURE 2. Measured concentrations for the 30 most frequently detected organic wastewater contaminants. Boxplots show concentration distribution truncated at the reporting level. Estimated values below the reporting level are shown. Estimated maximum values for coprostanol and cholesterol obtained from Method 5 (Table 1) are not shown. The analytical method number is provided in parentheses at the end of each compound name. An explanation of a boxplot is provided in Figure 1.

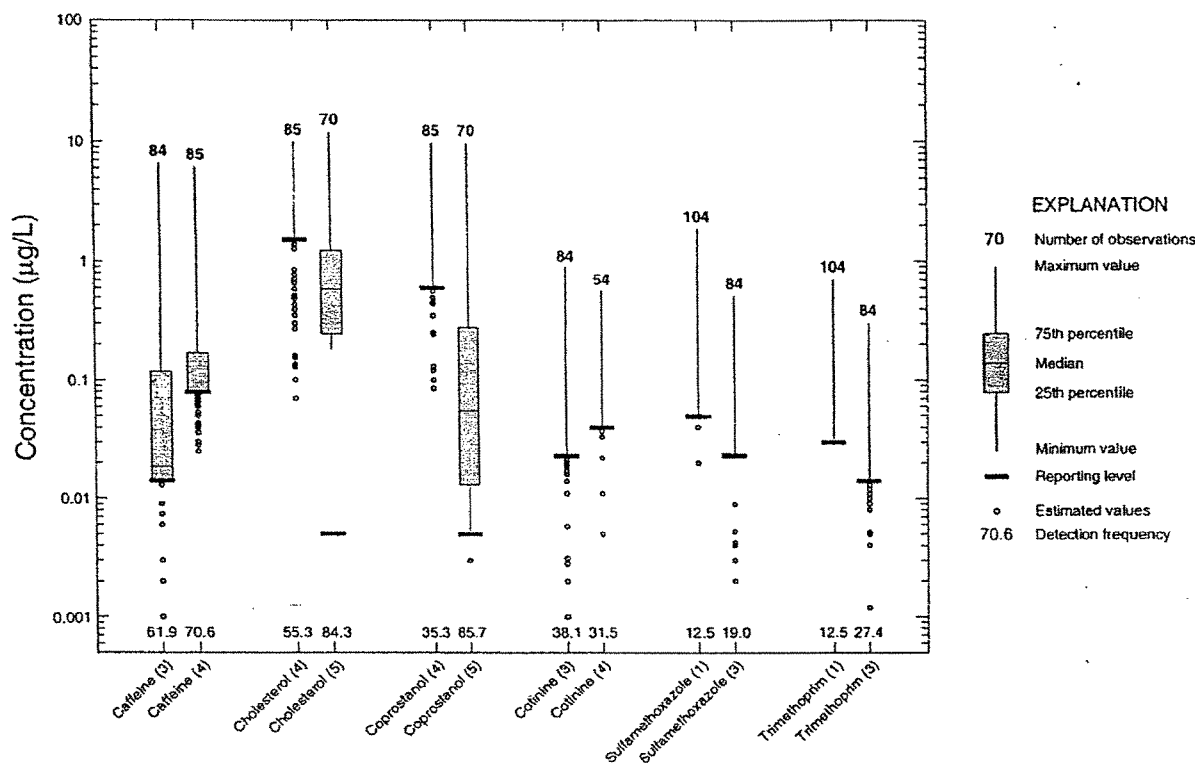


FIGURE 3. Comparison of concentrations of select compounds that were measured using two different methods with significantly different reporting levels. Boxplots show concentration distribution truncated at the reporting level. Estimated values below the reporting level are shown. Estimated maximum values for cholesterol and coprostanol obtained from Method 5 (Table 1) are not shown. The analytical method number is provided (in parentheses) at the end of each compound name.

Results and Discussion

One or more OWCs were found in 80% of the 139 streams sampled for this study. The high overall frequency of detection for the OWCs is likely influenced by the design of this study, which placed a focus on stream sites that were generally considered susceptible to contamination (i.e. downstream of intense urbanization and livestock production). In addition, select OWCs (such as cholesterol) can also be derived from nonanthropogenic sources. Furthermore, some of the OWCs were selected because previous research (28) identified them as prevalent in the environment. Thus, the results of this study should not be considered representative of all streams in the United States. A previous investigation of streams downstream of German municipal sewage treatment plants also found a high occurrence of OWCs (31).

A large number of OWCs (82 out of 95) were detected at least once during this study (Table 1). Only eight antibiotics and five other prescription drugs were not detected in the samples analyzed (Table 1). Measured concentrations were generally low (median detectable concentrations generally $<1 \mu\text{g/L}$, Table 1), with few compounds exceeding drinking-water guidelines, health advisories, or aquatic-life criteria (Table 1). The concentration of benzo[a]pyrene exceeded its maximum contaminant level (MCL) of $0.2 \mu\text{g/L}$ at one site and bis(2-ethylhexyl)phthalate concentrations exceeded its MCL of $6.0 \mu\text{g/L}$ at five sites. In addition, aquatic-life criteria were exceeded for chlorpyrifos (Table 1) at a single site. However, many of the 95 OWCs do not have such guidelines or criteria determined (Table 1). In fact, much is yet to be known about the potential toxicological effects of many of the OWCs under investigation (1). For many OWCs, acute effects to aquatic biota appear limited because of the low concentrations generally occurring in the environment (24, 32–34). More subtle, chronic effects from low-level envi-

ronmental exposure to select OWCs appear to be of much greater concern (1). Such chronic effects have been documented in the literature (34–38). In addition, because antibiotics are specifically designed to reduce bacterial populations in animals, even low-level concentrations in the environment could increase the rate at which pathogenic bacteria develop resistance to these compounds (15–17, 39).

The 30 most frequently detected compounds represent a wide variety of uses and origins including residential, industrial, and agricultural sources (Figure 2, Table 1). Only about 5% of the concentrations for these compounds exceeded $1 \mu\text{g/L}$. Over 60% of these higher concentrations were derived from cholesterol and three detergent metabolites (4-nonylphenol, 4-nonylphenol monoethoxylate, and 4-nonylphenol diethoxylate). The frequent detection of cotinine, 1,7-dimethylxanthine, erythromycin- H_2O , and other OWC metabolites demonstrate the importance of obtaining data on degradates to fully understand the fate and transport of OWCs in the hydrologic system. In addition, their presence suggests that to accurately determine the overall effect on human and environmental health (such as pathogen resistance and genotoxicity) from OWCs, their degradates should also be considered. The presence of the parent compound and/or their select metabolites in water resources has previously been documented for OWCs (40, 41) as well as other classes of chemicals such as pesticides (42, 43).

Many of the most frequently detected compounds (Figure 2) were measured in unfiltered samples using Method 4. Thus, their frequencies of detection may be somewhat higher because concentrations being measured include both the dissolved and particulate phases, whereas concentrations measured by Methods 1–3 include just the dissolved phase. For example, about 90% of the coprostanol discharged from

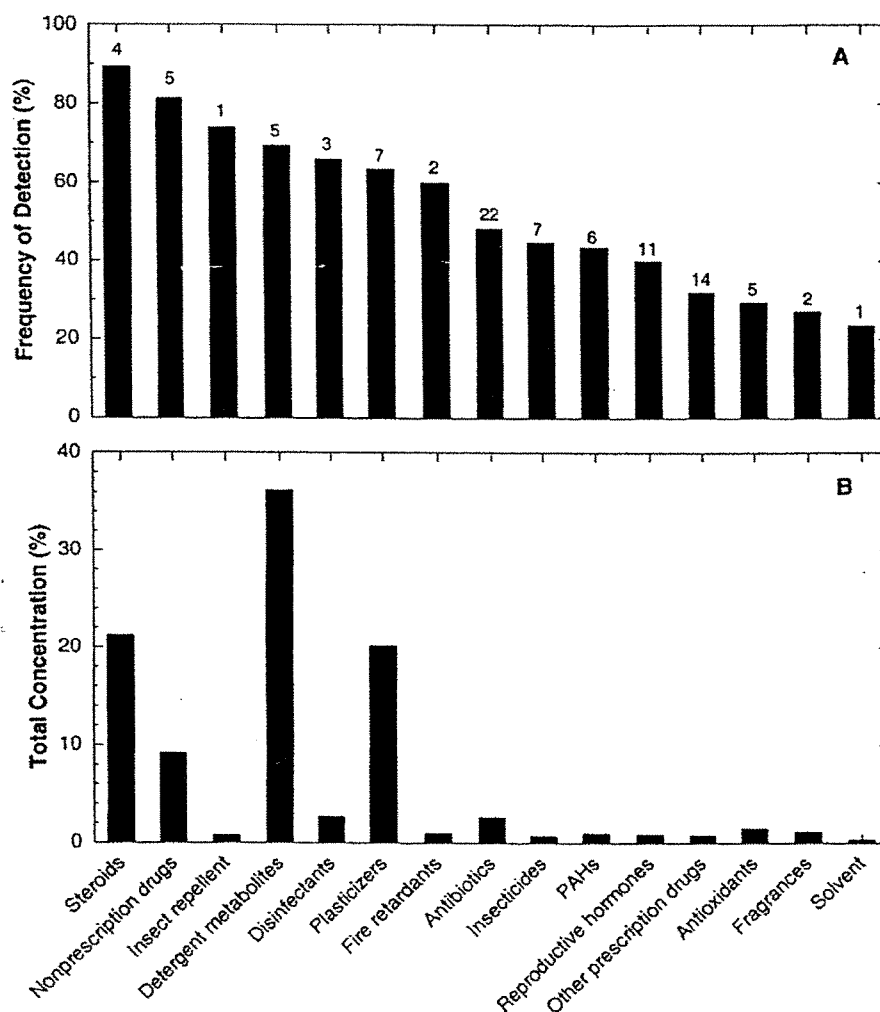


FIGURE 4. Frequency of detection of organic wastewater contaminants by general use category (4A), and percent of total measured concentration of organic wastewater contaminants by general use category (4B). Number of compounds in each category shown above bar.

sewage effluents has been shown to be associated with particulate matter (44). Thus, the concentration and frequency of detection for select compounds would likely have been reduced if sample filtration had taken place.

Variations in RL also influence the frequency of OWC detection (Figure 2). For example, the detection of 4-nonylphenol would likely have been much greater if an order of magnitude lower RL (similar to other OWCs) could have been achieved. The effect of RL on frequencies of detection is more clearly demonstrated by comparison of concentrations of select compounds that were measured using multiple analytical methods (Figure 3). As expected, the frequency of detection for a given compound was higher with the lower RL. The only exception being caffeine, where filtration of Method 3 may have reduced caffeine concentrations compared to that of the unfiltered Method 4. Figures 2 and 3 also demonstrate the importance of estimated values (45) below the RL. Clearly the numerous estimated concentrations illustrate that the current RLs are not low enough to accurately characterize the total range of OWC concentrations in the stream samples and that the frequencies of detection for this study are conservative.

To obtain a broader view of the results for this study, the 95 OWCs were divided into 15 groups based on their general uses and/or origins. The data show two environmental

determinations: frequency of detection (Figure 4A) and percent of total measured concentration (Figure 4B) for each group of compounds. These two views show a vastly different representation of the data. In relation to frequency of detection, there were a number of groups that were frequently detected, with seven of the 15 groups being found in over 60% of the stream samples (Figure 4A). However, three groups (detergent metabolites, plasticizers, and steroids) contributed almost 80% of the total measured concentration (Figure 4B).

For those groups of compounds that have received recent public attention—namely antibiotics, nonprescription drugs, other prescription drugs, and reproductive hormones (1, 2, 10)—nonprescription drugs were found with greatest frequency (Figure 4A). Antibiotics, other prescription drugs, and reproductive hormones were found at relatively similar frequencies of detection. The greater frequency of detection for nonprescription drugs may be at least partially derived from their suspected greater annual use compared to these other groups of compounds. When toxicity is considered, measured concentrations of reproductive hormones may have greater implications for health of aquatic organisms than measured concentrations of nonprescription drugs. Previous research has shown that even low-level exposure ($<0.001 \mu\text{g/L}$) to select hormones can elicit deleterious effects in aquatic species (7, 46, 47).

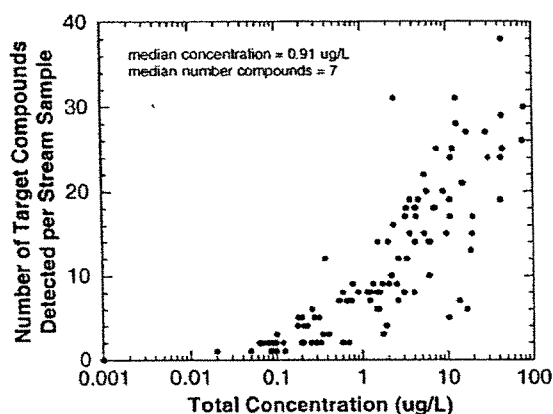


FIGURE 5. Relation between total concentration (summation from all detections) and number of organic wastewater contaminants found per water sample (Spearman's rank correlation coefficient = 0.94, $P < 0.001$).

Mixtures of various OWCs were prevalent during this study, with most (75%) of the streams sampled having more than one OWC identified. In fact, a median of seven OWCs were detected in these streams, with as many as 38 compounds found in a given streamwater sample (Figure 5). Because only a subset of the 95 OWCs were measured at most sites collected during the first year of study, it is suspected that the median number of OWCs for this study is likely underestimated. Although individual compounds were generally detected at low-levels, total concentrations of the OWCs commonly exceeded 1 $\mu\text{g/L}$ (Figure 5). In addition, 33 of the 95 target OWCs are known or suspected to exhibit at least weak hormonal activity with the potential to disrupt normal endocrine function (4, 7, 8, 10, 12, 22, 36, 37, 48–50), all of which were detected in at least one stream sample during this study (Table 1). The maximum total concentration of hormonally active compounds was 57.3 $\mu\text{g/L}$. Aquatic species exposed to estrogenic compounds have been shown to alter normal hormonal levels (7, 48, 51). Thus, the results of this study suggest that additional research on the toxicity of the target compounds should include not only the individual OWCs but also mixtures of these compounds. The prevalence of multiple compounds in water resources has been previously documented for other contaminants (52, 53). In addition, research has shown that select chemical combinations can exhibit additive or synergistic toxic effects (54–56), with even compounds of different modes of action having interactive toxicological effects (57).

The results of this study document that detectable quantities of OWCs occur in U.S. streams at the national scale. This implies that many such compounds survive wastewater treatment (1, 6, 58) and biodegradation (59). Future research will be needed to identify those factors (i.e. high use and chemical persistence) that are most important in determining the occurrence and concentration of OWCs in water resources.

Although previous research has also shown that antibiotics (60), other prescription drugs (1, 2, 19, 61–63), and non-prescription drugs (1, 40, 62, 64) can be present in streams, this study is the first to examine their occurrence in a wide variety of hydrogeologic, climatic, and land-use settings across the United States. Much is yet to be learned pertaining to the effects (particularly those chronic in nature) on humans, plants, and animals exposed to low-level concentrations of pharmaceuticals and other OWCs. Furthermore, little is known about the potential interactive effects (synergistic or antagonistic toxicity) that may occur from complex mixtures of these compounds in the environment. Finally,

additional research also needs to be focused on those OWCs not frequently detected in this stream sampling. Select OWCs may be hydrophobic and thus may be more likely to be present in stream sediments than in streamwater (65, 66). For example, the low frequency of detection for the tetracycline (chlortetracycline, doxycycline, oxytetracycline, tetracycline) and quinolone (ciprofloxacin, enrofloxacin, norfloxacin, sarafloxacin) antibiotics is not unexpected given their apparent affinity for sorption to sediment (66). In addition, select OWCs may be degrading into new, more persistent compounds that could be transported into the environment instead of (or in addition to) their associated parent compound.

Acknowledgments

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Pharmaceuticals and personal care products (PPCPs) in surface and treated waters of Louisiana, USA and Ontario, Canada

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Abstract

A newly developed analytical method was used to measure concentrations of nine pharmaceuticals and personal care products (PPCPs) in samples from two surface water bodies, a sewage treatment plant effluent and various stages of a drinking water treatment plant in Louisiana, USA, and from one surface water body, a drinking water treatment plant and a pilot plant in Ontario, Canada. The analytical method provides for simultaneous extraction and quantification of the following broad range of PPCPs and endocrine-disrupting chemicals: naproxen; ibuprofen; estrone; 17 β -estradiol; bisphenol A; chlorophene; triclosan; fluoxetine; and clofibric acid. Naproxen was detected in Louisiana sewage treatment plant effluent at 81–106 ng/l and Louisiana and Ontario surface waters at 22–107 ng/l. Triclosan was detected in Louisiana sewage treatment plant effluent at 10–21 ng/l. Of the three surface waters sampled, clofibric acid was detected in Detroit River water at 103 ng/l, but not in Mississippi River or Lake Pontchartrain waters. None of the other target analytes were detected above their method detection limits. Based on results at various stages of treatment, conventional drinking-water treatment processes (coagulation, flocculation and sedimentation) plus continuous addition of powdered activated carbon at a dosage of 2 mg/l did not remove naproxen from Mississippi River waters. However, chlorination, ozonation and dual media filtration processes reduced the concentration of naproxen below detection in Mississippi River and Detroit River waters and reduced clofibric acid in Detroit River waters. Results of this study demonstrate that existing water treatment technologies can effectively remove certain PPCPs. In addition, our study demonstrates the importance of obtaining data on removal mechanisms and byproducts associated with PPCPs and other endocrine-disrupting chemicals in drinking water and sewage treatment processes.

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1. Introduction

Recent studies indicate the potential widespread occurrence of low-level concentrations (ng– μ g/l)

of pharmaceuticals, hormones, and other organic sewage contaminants and their metabolites in the aquatic environment (Guillette, 1995; Desbrow et al., 1998; Halling-Sørensen et al., 1998; Ternes, 1998; Daughton and Ternes, 1999; Sedlak et al., 2000; Boyd and Grimm, 2001; Kolpin et al.,

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Table 1
Target analytes

Name	Trade name examples	CAS#	Purity (%)	Stock concentration (mg/l)	Commercial use	Chemical name
Clofibric acid	NA	882-09-7	97.0	10.17	Metabolite of lipid regulator	2-(4-Chlorophenoxy)-2-methylpropanoic acid
Naproxen	Naprosyn, Aleve	22204-53-1	100.6	10.02	Anti-inflammatory, analgesic	(α S)-6-Methoxy- α -methyl-2-naphthaleneacetic acid
Ibuprofen	Advil, Motrin	15687-27-1	99.8	30.08	Anti-inflammatory	α -Methyl-4-(2-methylpropyl)benzene-acetic acid
Acetaminophen	Tylenol	103-90-2	> 99.0	100.08	Analgesic	N-(4-Hydroxyphenyl)acetamide
Caffeine	Caffeine	58-08-2	> 99.9	99.9	Stimulant	3,7-Dihydro-1,3,7-trimethyl-1H-purine-2,6-dione
Fluoxetine ^a	Prozac	54910-89-3	100.0	357.6	Antidepressant	N-Methyl- γ -[4-(trifluoromethyl)phenoxy]benzenepropanamine
Chlorophene	Santophen I	120-32-1	NA	5.08	Disinfectant	4-Chloro-2-(phenylmethyl)phenol
Triclosan	Ster-Zac	3380-34-5	97.0	5.06	Antibacterial, disinfectant	5-Chloro-2-(2,4-dichlorophenoxy)phenol
Bisphenol A	Bisphenol A	80-05-7	> 99.0	5.11	Plastics intermediate, fungicide	4,4'-(1-Methylethylidene)bisphenol
Estrone	Estrol, Femidyn	53-16-7	> 99.0	10.14	Steroid	3-Hydroxyestra-1,3,5(10)-trien-17-one
17 β -Estradiol	Estrace, Estraderm	50-28-2	> 98.0	9.99	Steroid	(17 β)-Estra-1,3,5(10)-triene-3,17-diol

All chemicals were obtained from Sigma Chemical Corporation (St. Louis, MO). Stock concentrations were prepared in dichloromethane. NA, not available.

^a Purchased as fluoxetine hydrochloride.

2002). Many of these compounds are suspected or potential endocrine-disrupting chemicals. Pharmaceuticals and personal care products (PPCPs) describe a large class of chemical contaminants that can originate from human usage and excretions, and veterinary applications of a variety of products, such as over-the-counter and prescription medications, and fungicides and disinfectants used for industrial, domestic, agricultural and livestock practices (Daughton and Ternes, 1999). PPCPs and their metabolites are continually introduced into aquatic environs and are prevalent at detectable concentrations (Kolpin et al., 2002), which can affect water quality and ecosystem health and potentially impact drinking water supplies (Roefler et al., 2000; Trussell, 2001). The long-term effects of continuous, low-level exposure to PPCPs and their metabolites are not well understood (Daughton and Ternes, 1999).

Effluents from sewage treatment plants contain a variety of PPCPs (Daughton and Ternes, 1999). Studies have shown that the transformation pro-

cesses for specific PPCP compounds can vary in a sewage treatment plant, depending on the characteristics of the sewage, weather conditions, and the design and operation of the treatment processes (Ternes, 1998; Johnson and Sumpter, 2001). Upon discharge of treated sewage into a receiving water body, residual PPCPs can be diluted and blended with contaminants from other discharge points, as well as runoff and seepage. These same receiving water bodies also can serve as drinking water sources. Recent studies aimed at investigating drinking-water treatment methods for PPCPs have demonstrated that conventional treatment processes (coagulation, flocculation and sedimentation) are not effective methods, but other treatment processes, such as oxidation with chlorine and ozone, activated carbon and membrane filtration, can be effective in removing antibiotics (Adams et al., 2002) and other selected pharmaceuticals (Ternes et al., 2002).

A list of target analytes representing a variety of PPCPs was developed for this study (Table 1).

To date, there is no universally accepted method for the analysis of PPCPs in aquatic environs. Several analytical approaches have been utilized, including gas chromatography/mass spectrometry (GC/MS), gas chromatography/mass spectrometry/mass spectrometry (GC/MS/MS), GC with high-resolution mass spectrometry, liquid chromatography-ultraviolet detection (LC-UV), liquid chromatography/mass spectrometry (LC/MS) and liquid chromatography/mass spectrometry/mass spectrometry (LC/MS/MS) (Desbrow et al., 1998; Barber et al., 2000; Laganà et al., 2000; Möder et al., 2000; Huang and Sedlak, 2001; Kolpin et al., 2002). The decision to use GC or LC is usually based on the physicochemical characteristics of the target analyte. Many PPCPs contain polar functional groups and/or are thermally labile and do not lend themselves readily to GC analysis. Thus, most GC methods for polar PPCPs must incorporate a derivatization step to overcome these limitations. LC is generally applicable to more polar compounds, in contrast to GC. The use of MS for detection in either case gives a second dimension of information, which can be used to confirm the identity of the targeted compound through its mass spectrum. Nonetheless, PPCPs include a broad range of molecules of differing polarity and functionality, and hence pose a significant analytical challenge.

The objective of this study was to develop a method for quantifying the concentration of a target list of a variety of PPCPs in surface and treated waters of Louisiana, USA and Ontario, Canada. This paper provides a method for analyzing the following diverse list of PPCP compounds in natural and treated water samples: a metabolite (clofibric acid) of a lipid regulator; two analgesics (naproxen and ibuprofen); two steroids (estrone and 17β -estradiol); a chemical intermediate in the synthesis of commercial products (bisphenol A); one disinfectant (chlorophene); an antibacterial additive (triclosan); and an antidepressant (fluoxetine). The target list of PPCPs is inclusive of bisphenol A, an intermediate, due to its ubiquitous nature and its endocrine-disrupting potential. The method was used to determine concentrations of the target PPCPs in surface water samples from the Mississippi River and Lake Pontchartrain in Louisiana, and the Detroit River in Ontario. The

method was also used to analyze treated water samples from a sewage treatment plant in Louisiana, several stages of drinking water treatment plants in Louisiana and Ontario, and a pilot drinking-water treatment plant in Ontario.

2. Site selection and sampling

Surface water samples were collected from the Mississippi River in New Orleans, Louisiana during September–November 2001 (Fig. 1, Site #1). The Mississippi River extends from northern Minnesota to the Gulf of Mexico and drains 41% of the conterminous United States in an area where 27% of the population resides (Meade, 1996). The mean annual discharge of the lower Mississippi River near New Orleans is $13\,500\text{ m}^3/\text{s}$ (Meade, 1996). The Mississippi River receives a variety of organic wastes from urban areas, farms, factories and individual households. Approximately 70 US cities rely on the Mississippi River as a source of drinking water. Surface water samples were collected from the Mississippi River at a site outside of direct influence of discharge points of known private or municipal sewage treatment plants.

Surface water samples were also collected on the southern shore of Lake Pontchartrain (Fig. 1, Site #2), which is located within the Lake Pontchartrain estuary in the central Gulf Coast region adjacent to New Orleans, Louisiana. Lake Pontchartrain is influenced by riverine discharges ($228\text{ m}^3/\text{s}$) as well as stormwater drainage and freshwater diversion from the Mississippi River through the Bonnet Carre spillway (Flowers and Isphording, 1990; Argyrou et al., 1997). Lake Pontchartrain is not used as a municipal drinking water source.

Sewage plant effluent samples were collected during February and March 2002 from the Jefferson Parish East Bank Wastewater Treatment Plant (Fig. 1, Site #3), which discharges treated sewage effluent into the Mississippi River. The plant is located in metropolitan New Orleans, Louisiana, approximately 5 km west of the city line. The plant uses conventional secondary wastewater treatment and operates at an annual average flow of $125\,000\text{ m}^3/\text{day}$. Treated sewage samples were collected prior to chlorination of the effluent (STP1), as shown in Fig. 2a.

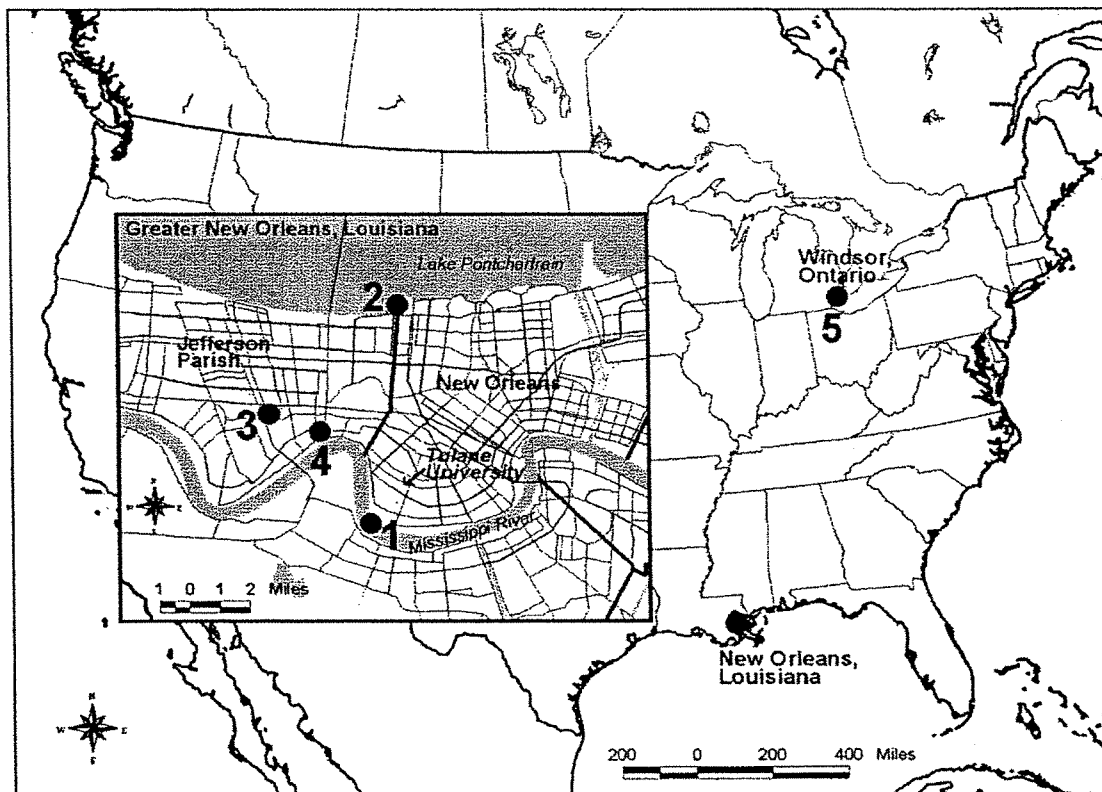


Fig. 1. Sampling sites in greater New Orleans, Louisiana and Windsor, Ontario. Site #1, Mississippi River, Louisiana; Site #2, Lake Pontchartrain, Louisiana; Site #3, Jefferson Parish East Bank Wastewater Treatment Plant, Louisiana; Site #4, Jefferson Parish East Bank Water Treatment Plant, Louisiana; Site #5, A.H. Weeks Water Treatment Plant and ENWIN Pilot Plant, Ontario.

Water samples were collected from various stages of the Jefferson Parish East Bank Water Treatment Plant (Fig. 1, Site #4), which relies on the Mississippi River as its source. The drinking water treatment plant is located approximately 2.5 km west of the New Orleans city line. The plant operates at a maximum flow of 330 000 m³/day and uses conventional treatment, which includes coagulation (alum and cationic polyelectrolyte polymer), flocculation and sedimentation. The treated water is disinfected by chlorination prior to filtration, and chloramination prior to distribution (Fig. 2b). High-load organic pollutants are removed from the raw water by adding powdered activated carbon (PAC) at a concentration of 2 mg/l. Samples were collected at the plant inlet

(JP1), after PAC addition and conventional treatment (JP2), and after chlorination, filtration and storage (JP3), as shown in Fig. 2b.

In Canada, water samples were collected in January 2002 at the A.H. Weeks Water Treatment Plant (Fig. 1, Site #5) in Windsor, Ontario, which relies on the Detroit River as its source. The drinking water treatment plant operates at a maximum flow of 227 000 m³/day and uses ozonation, conventional treatment (alum and Percol LT22 as coagulants) and chlorination prior to distribution (Fig. 2c). Samples were collected from the Detroit River at the plant inlet (WO1) and after treatment at the plant outlet (WO2), as shown in Fig. 2c. Samples were also collected at the ENWIN Pilot Plant (Fig. 2d), which was located at the same

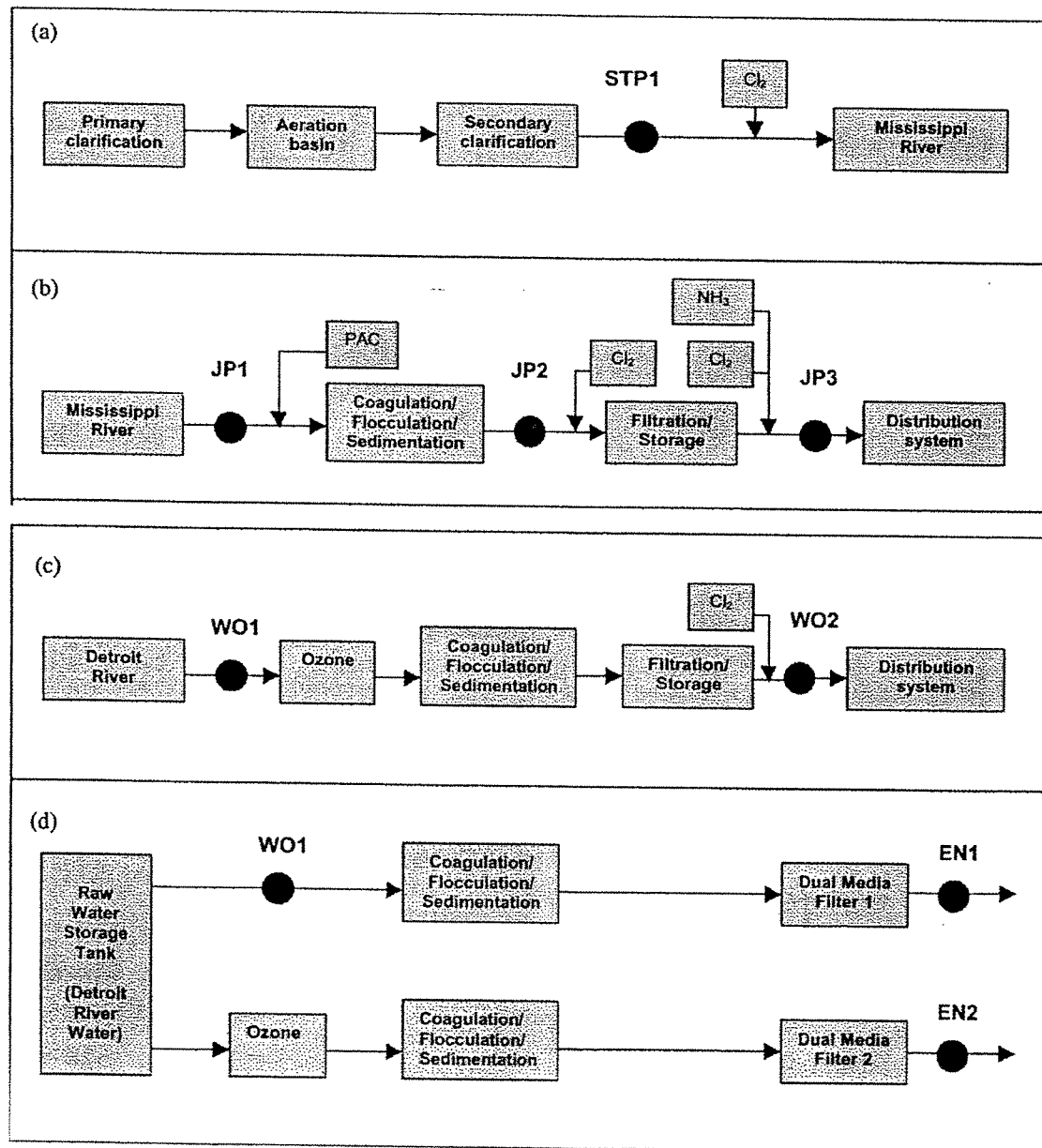


Fig. 2. Process flow diagrams for sewage and drinking water treatment plants in Louisiana and Ontario (● indicates sampling location): (a) Jefferson Parish East Bank Wastewater Treatment Plant, Louisiana (Fig. 1, Site #3); (b) Jefferson Parish East Bank Water Treatment Plant, Louisiana (Fig. 1, Site #4); (c) A.H. Weeks Water Treatment Plant, Ontario (Fig. 1, Site #5); and (d) ENWIN Pilot Plant, Ontario (Fig. 1, Site #5).

site as the A.H. Weeks Water Treatment Plant and used to test treatment processes for the Detroit River water source (WO1). Water samples were collected at the outlet of the pilot plant following conventional treatment and dual media filtration (EN1) and at the outlet of a similar process train preceded by ozonation (EN2), as shown in Fig. 2d.

For all sites, a total of 8 l was collected as grab samples using pre-cleaned 4-l amber glass containers. Louisiana samples were stored on ice during transport to the laboratory and were processed within 7 days. Samples collected from the Canadian plants were acidified prior to shipping and were analyzed immediately upon arrival at Tulane University. A method blank using ultra-pure laboratory water further purified by passing through a SPE disk prior to use (see below) was performed for each batch of samples collected from the sewage and drinking water treatment plants.

3. Analytical methods

A relative response factor (RRF) standard solution of all reference standards was prepared in dichloromethane (DCM) and methanol, with concentrations of each analyte ranging between 5 and 100 mg/l. The RRF standard consisted of the compounds summarized in Table 1 (except fluoxetine hydrochloride, which was prepared separately) and three deuterated surrogate compounds (bisphenol A- d_{14} , estrone- d_4 and acetaminophen- d_4). Fluoxetine was not as stable as our other target analytes; therefore, fluoxetine standards were prepared from the solid material just prior to use. The surrogate standard was prepared in DCM at concentrations between 5 and 100 mg/l prior to adding it to the RRF standard. Phenanthrene- d_{10} (99.3% purity, AccuStandard Inc, New Haven, CT) was chosen as the internal standard, since it was not affected by the derivatization step in the analytical procedure, as discussed below. Phenanthrene- d_{10} was prepared in DCM at a concentration of 495.4 mg/l and added to the sample after the final concentration step and after derivatization.

Sampling bottles and all glassware used for sample collection and preparation were cleaned by washing with soap, soaking in a 5% Contrad

solution (Decon Laboratories Inc, Bryn Mawr, PA) and in hydrochloric acid (2 N), and then ashing at 450 °C. All laboratory materials were either made of glass or Teflon to avoid sample contamination. Teflon containers were cleaned in the same manner as glassware, but without ashing. Ultra-pure water was produced in the laboratory by filtering tap water through activated carbon, followed by a mixed-bed deionization tank and ultra-filtration membrane system, and then ultraviolet light exposure (US Filter, Modulab UF/UV, CA, USA). Analysis of ultra-pure water used for spiked recovery experiments and method blanks showed low-level background contamination with bisphenol A. Once this was determined, the procedure was modified to include further purification of the ultra-pure water by passing it through a SPE disk. All solvents were GC grade.

3.1. Solid-phase extraction

The targeted PPCP compounds were isolated from water samples by solid-phase extraction using a polar SDB-XC Empore disk (3M Corporation, St. Paul, MN). Surface water samples, sewage treatment plant effluent samples and untreated drinking water treatment plant samples were pumped through 1.0- and 0.2- μ m glass fiber filters (47 mm in diameter, Millipore Corporation, Bedford, MA) to remove particulate matter prior to solid-phase extraction. Pre-filtration was not necessary for water samples collected at the outlet of the drinking water treatment plants. Extraction disks were pre-conditioned with 50 ml of methanol, 50 ml of DCM, 50 ml of methanol and 10 ml of ultra-pure water. If samples were not previously acidified, the pH was adjusted to <2.0 using 12 N HCl prior to spiking with the surrogate standard (0.5 ml/l sample). Samples were then drawn through the extraction disks using vacuum aspiration at an approximate flow rate of 100 ml/min. The disks were then air-dried and the targeted compounds were extracted from the disks by eluting with 50 ml of methanol, 50 ml of DCM and 50 ml of methanol. The extracts were concentrated to an approximate volume of 1 ml using a RapidVap® with mild heat (50 °C) and a gentle stream of nitrogen gas.

The concentrated organic extracts were passed through a column containing 3 g of pre-washed silica gel to remove dissolved interfering compounds (e.g. humic acids) from some samples. The silica gel was then washed with three bed volumes each of DCM and methanol. This clean-up step was added to the analytical procedure after a method revision in the course of this research, and therefore was not applied to all samples. The silica gel-treated samples were carefully evaporated to a volume of 1 ml under the same conditions described previously.

3.2. Derivatization

Derivatization was used to enhance the thermal stability of clofibric acid, which thermally degraded in the GC injection port, and reduce the polarity of specific target analytes (clofibric acid, ibuprofen and naproxen) to facilitate GC analysis. Given the sensitivity of the derivatization reagent [*N,O*-bis(trimethylsilyl)-trifluoroacetamide in the presence of trimethylchlorosilane; BSTFA; Supelco Inc, Bellefonte, PA] to moisture, and because Na_2SO_4 was not effective at removing traces of water dissolved in methanol, all samples were placed in GC autosampler vials and completely dried under a stream of N_2 prior to derivatization. Derivatization was achieved by dissolving the dried sample residue in 1 ml of BSTFA reagent mixture. The closed vial was then heated at 80 °C for 20 min. Finally, 10 μl of the internal standard (phenanthrene- d_{10}) was added to the sample prior to instrumental analysis.

For the RRF and instrument detection limit experiments, the working standards were carefully dried, dissolved in the BSTFA reagent mixture and derivatized as described here. A 1-ml aliquot of known concentration was prepared for each target compound and analyzed by GC/MS. This same sample was then dried and derivatized as described previously. The derivatized sample was analyzed by GC/MS and the chromatogram was checked for both the non-derivatized and derivatized forms of the analyte. If the derivatization was incomplete, the percentage completion was determined by comparing the peak areas. Caffeine and fluoxetine, lacking the appropriate functional groups, exhibit-

ed no response to derivatization. Estrone was derivatized to 84.7% completion. All other analytes were derivatized to 100% completion.

3.3. GC/MS conditions

Samples were analyzed by GC/MS (Agilent 6890 GC and 5972 MSD) under the following conditions. Splitless 2- μl injections were made onto a DB-5MS column (25 m with 0.25- μm film thickness and 0.25 mm i.d.) at a constant flow rate of 1 ml/min. The GC oven was operated from 100 °C (0-min hold) at 5 °C/min to 165 °C (5-min hold), then at 2 °C/min to 175 °C (0-min hold) and at 10 °C/min to 320 °C (5-min hold) for a total run time of 42.5 min. The injector and detector temperatures were 230 and 300 °C, respectively. The MS was operated in +EI mode using selected ion monitoring (SIM) for sensitivity. Table 2 summarizes the SIM conditions.

3.4. Quantification

Quantification of the targeted PPCP compounds was conducted by comparing peak areas of the most intensive ion of each compound with that of the internal standard. Compound identification was confirmed by GC retention time and qualifier ions (usually molecular ion and one or two fragment ions) as shown in Table 2. Baseline interference was observed at or near the retention time of estrone. As part of the method development, ion ratios were monitored, enabling discrimination between interference and the proper response for estrone. In addition, qualifier ions were re-evaluated for the steroid compounds as compared to methods employed by the authors in previous research (Boyd and Grimm, 2001). Before each sequence of samples, response factors were calculated separately from the analysis of the RRF and its dilutions, 1:10, 1:20 and 1:200.

Fig. 3a shows the GC/MS chromatogram of the RRF stock solution containing the target compounds. Fig. 3b shows the chromatogram of a sample collected from the inlet of Jefferson Parish East Bank Water Treatment Plant representing raw Mississippi River water (Fig. 2b, JP1). It identifies

Table 2
Selected ion monitoring (SIM) program for targeted and standard analytes

SIM group	Type	Name	Molecular weight	Retention time (min)	Target ion	Qualifier ion	
						1	2
1	TGT	Clofibric acid–TMS	286	17.66	128	143	286
1	TGT	Ibuprofen–TMS	278	19.61	263	278	234
2	SS	Acetaminophen–d ₄ –TMS	227	ND	227	–	–
2	SS	Acetaminophen–d ₄ –TMS(2)	299	19.79	284	299	–
2	TGT	Acetaminophen–TMS(2)	295	19.83	280	295	206
3	IS	Phenanthrene–d ₁₀	188	26.11	188	160	–
3	TGT	Caffeine	194	27.30	194	109	–
3	TGT	Fluoxetine	309	ND	309	104	–
4	TGT	Clorophene–TMS	290	28.39	290	292	275
4	TGT	Naproxen–TMS	302	30.77	243	302	185
5	TGT	Triclosan–TMS	360	31.33	200	360	362
6	SS	Bisphenol A–d ₁₆	244	ND	226	–	–
6	SS	Bisphenol A–d ₁₅ –TMS(1)	315	ND	315	–	–
6	SS	Bisphenol A–d ₁₄ –TMS(2)	386	32.08	368	386	–
6	TGT	Bisphenol A–TMS(2)	372	32.17	357	372	–
7	SS	Estrone–d ₄ –TMS	346	36.21	346	220	–
7	TGT	Estrone–TMS	342	36.21	342	327	257
7	TGT	Estrone–d ₄	274	ND	274	–	–
8	TGT	17 β -Estradiol–TMS(2)	416	36.43	416	285	–

IS, internal standard; ND, not detected; SS, surrogate standard; TGT, targeted analyte; TMS, Trimethylsilyl derivative. Note that (1) or (2) after TMS refers to the mono- or di-derivative, respectively.

the response of the target ion and two qualifier ions of naproxen.

3.5. Limits of detection and determination

The instrument detection limits for all compounds were determined by serial dilution of the RRF and fluoxetine standard solutions. The diluted solutions were prepared by weighing a known amount of working standard into an autosampler crimp-top vial and adding a known mass of BSTFA derivatization reagent and a known amount of internal standard. In this way, the injected mass of each compound could be calculated. Instrument detection limits are reported in Table 3.

3.6. Recoveries

Natural water samples were collected from three surface water bodies, and treated water samples were collected from the effluent of a sewage treatment plant and various stages of treatment from two drinking water treatment plants and a

pilot plant. As such, sample matrices were diverse and surrogate standards were added to samples to monitor matrix effects. Spiked recoveries were measured for each compound, including surrogates using ultra-pure laboratory water. Three 1-l laboratory samples were spiked with 1 ml each of a RRF and a fluoxetine standard. The spiked samples were extracted and analyzed using solid-phase extraction, derivatization and GC/MS as described previously. Results were compared to non-extracted RRF and fluoxetine standard solutions. For quantification, the samples and the standard solution were spiked with 10 μ l of the internal standard.

Recoveries for most compounds were greater than 47%. Exceptions were acetaminophen and caffeine. Acetaminophen was repeatedly not detected, whereas caffeine exhibited low recovery (2.8%). These low recoveries were attributed to incomplete retention of these compounds on the extraction disk. Recovery rates for the surrogate compounds bisphenol A–d₁₆ and estrone–d₄ were greater than 95%. The recovery rate for acetamin-

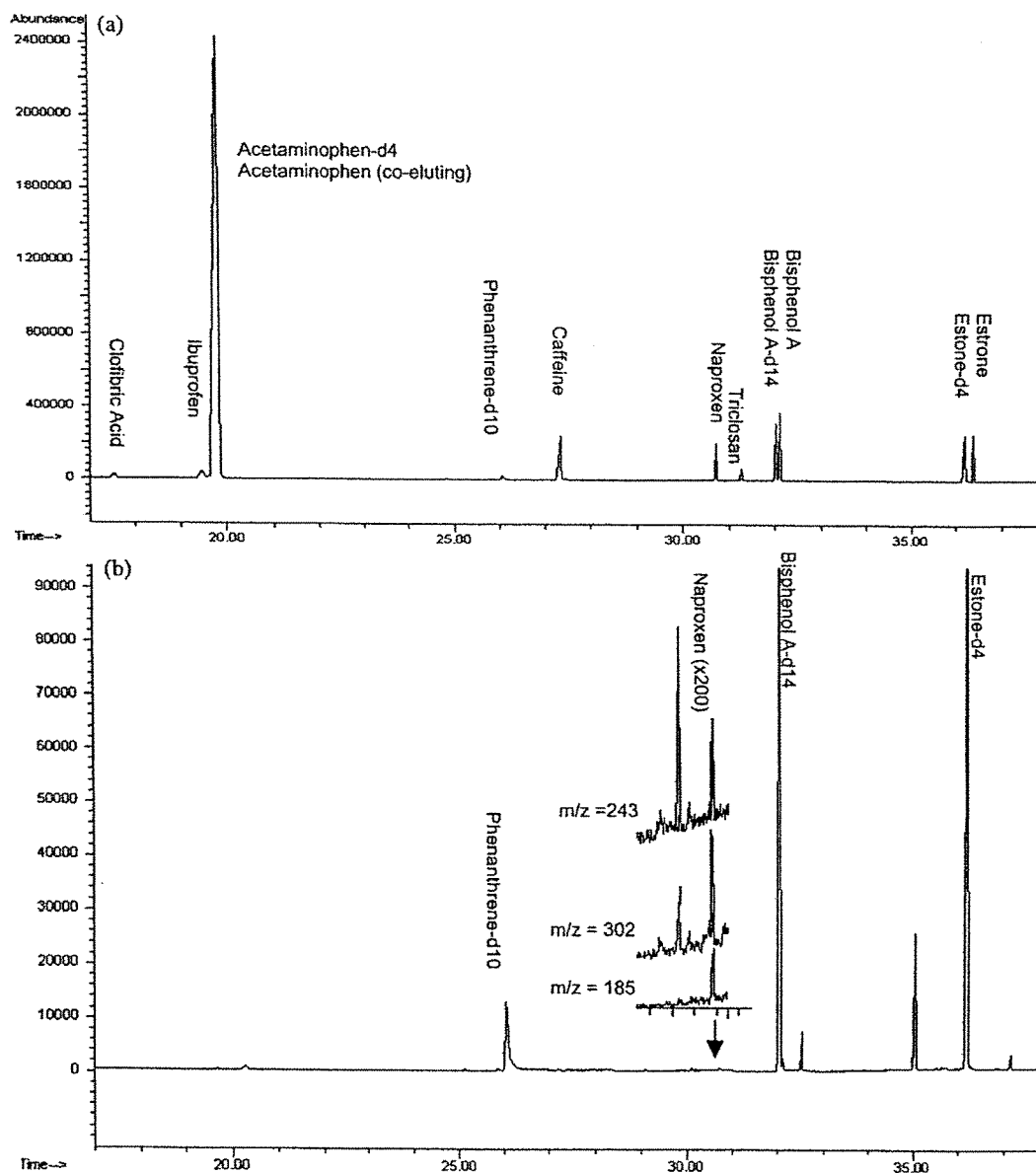


Fig. 3. GC/MS chromatograms: (a) relative response factor standard containing all target compounds (except fluoxetine and chlorophene); (b) inlet sample (JP1) for Jefferson Parish East Bank Water Treatment Plant (Site #4, Fig. 1).

open-d₄ could not be determined, as GC/MS analysis repeatedly showed non-detection for this compound. Acetaminophen and caffeine were therefore not included in the final list of target analytes for this method. Recovery rates and meth-

od detection limits for the target compounds are summarized in Table 3.

In contrast to spiked recovery experiments with ultra-pure water, for which the recovery was greater than 95% for estrone-d₂ and bisphenol A-d₁₄,

recoveries of deuterated compounds were slightly decreased in most surface water samples, which was attributed to the presence of high loads of dissolved organic matter and other matrix complexities. Samples collected from the Louisiana sewage treatment plant and the Louisiana drinking-water treatment plant were filtered through a silica gel column to remove dissolved organic matter. This clean-up step was not applied to samples from the Canadian plants and it had no significant effect on recovery of the target analytes. Water samples that were disinfected with chlorine at the drinking water treatment plants were quenched with 40–50 mg/l of sodium sulfite to avoid reaction of chlorine residuals with the surrogate standard compounds. Samples that were stored in the refrigerator for several days exhibited improved surrogate standard recovery, which was attributed to the dissipation of free chlorine prior to sample analysis.

4. Results and discussion

Sampling results for the nine targeted PPCP compounds are summarized in Table 4 Tables 5–

Table 4
PPCPs in surface waters in Louisiana

PPCP compound	Concentration in surface water (ng/l)			
	Mississippi River		Lake Pontchartrain	
Clofibric acid	ND	ND	ND	ND
Ibuprofen	ND	ND	ND	ND
Fluoxetine	ND	ND	ND	ND
Chlorophene	ND	ND	ND	ND
Naproxen	37	39	107	22
Triclosan	ND	ND	ND	ND
Bisphenol A	NQ	NQ	NQ	NQ
Estrone	ND	ND	ND	ND
17 β -Estradiol	ND	ND	ND	ND
Bisphenol A-d ₁₄ (%) ^a	68.0	75.0	67.0	67.0
Estrone-d ₄ (%) ^a	103.4	119.4	88.3	124.6
Acetaminophen-d ₄ (%) ^a	ND	ND	ND	ND

Samples were collected from the shores of the Mississippi River (Fig. 1, Site #1) and from the shores of Lake Pontchartrain (Fig. 1, Site #2). No silica gel cleanup was used during sample preparation. ND, not detected (see MDLs in Table 3); NQ, not quantified.

^a Percentage recovery of surrogate standard.

Table 3
Detection limits and percentage recovery

	IDL (ng/l)	Completion of derivatization (%)	Method development			Revised method		
			MDL (ng/l)	Recovery (%)	R.S.D. (%)	MDL (ng/l)	Recovery (%)	R.S.D. (%)
Clofibric acid	3	100	0.6	60.8	12.6	0.8	44.2	26.5
Ibuprofen	13	100 ^b	3.5	47.1	26.9	2.6	63.0	12.3
Acetaminophen	45	100	ND	ND	ND	ND	ND	ND
Caffeine	24	0	107.1	2.8	3.6	319.3	0.9	1.1
Fluoxetine	178	0	25.8	86.1	7	25.4	87.7 ^a	–
Chlorophene	0.6	100	0.1	71.7	5.9	0.1	108.9 ^a	–
Naproxen	3	100	0.4	87.9	2.8	0.4	102.9	17.8
Triclosan	1	100	0.2	53.8	24	0.2	60.1	22.8
Bisphenol A	0.6	100	0.1	99.7	3.5	0.1	95.6	39.5
Estrone	3	84.7	0.4	91.9	5.1	0.3	130.3	22.3
17 β -Estradiol	1	100	0.1	90.5	9.1	0.1	117.6	14.8

Method development does not include silica gel clean-up. Revised method includes silica gel clean-up. Completion of derivatization was based on comparison of underivatized peak area and derivatized peak area. IDL, instrument detection limit; MDL, method detection limit; ND, not detected; R.S.D., relative standard deviation. MDL was based on a 2- μ l injection from a 1-ml extract of an 8-l sample. Percentage recovery is based on non-extracted RRF and fluoxetine standard.

^a Due to fast degradation of this compound, the value of only one sample was considered.

^b Assumed completion. Underivatized ibuprofen did not elute from the GC.

Table 5
PPCPs in sewage treatment plant effluent in Louisiana

PPCP compound	Concentration at STP1 (ng/l)	
Clofibric acid	ND	ND
Ibuprofen	ND	ND
Fluoxetine	ND	ND
Clorophene	ND	ND
Naproxen	106	81
Triclosan	21	10
Bisphenol A	ND	ND
Estrone	ND	ND
17 β -Estradiol	ND	ND
Bisphenol A-d ₁₄ (%) ^a	13.6	13.9
Estrone-d ₄ (%) ^a	52.6	28.9
Acetaminophen-d ₄ (%) ^a	1.1	1.2

Samples were collected at the Jefferson Parish East Bank Wastewater Treatment Plant (Fig. 1, Site #3). Sampling location is shown in Fig. 2a. Sample preparation included silica gel clean-up. ND, not detected (see MDLs in Table 3).

^a Percentage recovery of surrogate standard.

7. Results are discussed with regard to occurrence of these nine compounds in surface waters in Louisiana and Ontario, in the effluent of a sewage treatment plant, and during various stages of removal by drinking water treatment processes.

4.1. Surface waters

Results for Louisiana and Ontario surface waters are shown in Tables 4, 6 and 7. Naproxen, which is a common prescription pain reliever, was detected in Mississippi River (Table 4 and JP1 in Table 6), Lake Pontchartrain (Table 4) and Detroit River (WO1 in Table 7) waters at concentrations ranging from 22 to 107 ng/l. These observations are similar to findings reported by Ternes (1998) and Ternes et al. (1999) for German, Canadian and Brazilian surface waters. Clofibric acid, which is a metabolite of the lipid regulator clofibrate (as one of several in this class), was detected in Detroit River water (WO1 in Table 7) at a concentration of 103 ng/l, similar to findings for European surface waters (Stan et al., 1994; Stumpf et al., 1996; Ternes, 1998; Daughton and Ternes, 1999). The absence of clofibric acid in Mississippi River and Lake Pontchartrain waters could be attributed to the declining use of clofibrate in the United States (WHO, 1996).

17 β -Estradiol was observed to be below the method detection limit (Table 3) of 0.1 ng/l for all samples collected from surface waters. Other investigators have reported 17 β -estradiol in surface

Table 6
PPCPs at Jefferson Parish East Bank drinking water treatment plant in Louisiana, USA

PPCP compound	Concentration at water treatment plant (ng/l)					
	Mississippi R. (JP1)		Precipitator (JP2)		Finished water (JP3)	
Clofibric acid	ND	ND	ND	ND	ND	ND
Ibuprofen	ND	ND	ND	ND	ND	ND
Fluoxetine	ND	ND	ND	ND	ND	ND
Clorophene	ND	ND	ND	ND	ND	ND
Naproxen	64	65	63	68	ND	ND
Triclosan	ND	ND	ND	ND	ND	ND
Bisphenol A	NQ	NQ	NQ	NQ	NQ	ND
Estrone	ND	ND	ND	ND	ND	ND
17 β -Estradiol	ND	ND	ND	ND	ND	ND
Bisphenol A-d ₁₄ (%) ^a	62.8	65.2	46.0	81.3	94.9	18.6
Estrone-d ₄ (%) ^a	130.1	68.3	118.1	99.3	106.7	17.7
Acetaminophen-d ₄ (%) ^a	0.2	0.2	0.2	ND	0.1	ND

Samples were collected at Jefferson Parish East Bank Water Treatment Plant in Louisiana, USA (Fig. 1, Site #4). Sampling locations at the plant are shown in Fig. 2b. ND, not detected (see MDLs in Table 3). NQ, not quantified.

^a Percent recovery of surrogate standard. Sample preparation included silica gel clean-up.

Table 7
PPCPs at drinking water treatment plant and pilot plant in Ontario, Canada

PPCP compound	Concentration at water treatment plant (ng/l)			
	Full-scale plant		ENWIN pilot plant	
	Detroit R. water (WO1)	Finished water (WO2)	Filter 1 (EN1)	Filter 2 (EN2)
Clofibric acid	103	ND	ND	ND
Ibuprofen	ND	ND	ND	ND
Fluoxetine	ND	ND	ND	ND
Clorophene	ND	ND	ND	ND
Naproxen	63	ND	ND	ND
Triclosan	ND	ND	ND	ND
Bisphenol A	NQ	NQ	NQ	NQ
Estrone	ND	ND	ND	ND
17 β -Estradiol	ND	ND	ND	ND
Bisphenol A-d ₁₄ (%) ^a	66.7	93.6	80.2	91.5
Estrone-d ₄ (%) ^a	77.2	90.7	82.1	74.6
Acetaminophen-d ₄ (%) ^a	ND	0.2	ND	ND

Samples were collected at the A.H. Weeks Water Treatment Plant and ENWIN pilot plant in Ontario, Canada (Fig. 1, Site #5). Sampling locations are shown in Fig. 2c,d. Sample preparation did not include silica gel clean-up. ND, not detected (see MDLs in Table 3). NQ, not quantified.

^a Percentage recovery of surrogate standard.

waters at concentrations ranging from 0.2 to 2.6 ng/l (Snyder et al., 1999; Ternes et al., 1999). More data are therefore needed to determine the occurrence of 17 β -estradiol and other PPCPs at lower concentrations in Louisiana and Ontario surface waters.

Ibuprofen, fluoxetine, triclosan, estrone and 17 β -estradiol were not detectable in Mississippi River surface waters in our analyses. This observation is consistent with another study, which used multiple analytical techniques to determine PPCP target analytes (Barnes et al., 2002). Detectable but non-quantifiable levels of bisphenol A were found in several of our Mississippi River samples. In contrast, Barnes et al. (2002) were able to detect bisphenol A at a concentration of 60 ng/l in their analysis of Mississippi River surface waters. These contrasting results suggest a need to include bisphenol A as a target analyte in natural water samples.

4.2. Sewage treatment plant effluent

Results for samples collected from the effluent of the Louisiana sewage treatment plant (Table 5)

indicate naproxen at concentrations of 81 and 106 ng/l. This sewage treatment plant discharges effluent into the Mississippi River and these naproxen concentrations are approximately 2.5-fold greater than naproxen detected in Mississippi River water. Other investigators (Ternes, 1998; Stumpf et al., 1999) have reported similar findings for naproxen in wastewater effluent, ranging from 20 to 520 ng/l. Results of this study also indicate triclosan in the Louisiana sewage treatment plant effluent at concentrations ranging from 10 to 21 ng/l. Triclosan is added as an antibacterial agent to detergents and it has been reported in sewage treatment plant effluents at concentrations up to 650 ng/l (Paxéus, 1996; Lindström et al., 2002). For this study, samples were collected prior to chlorination of the effluent at the sewage treatment plant. As such, results from this study do not necessarily indicate the quality of the final treated water as discharged into the Mississippi River. Results from this Tulane study also indicate that no other targeted PPCPs were detected in the effluent from the sewage treatment plant.

4.3. Drinking water treatment processes

Samples collected at the inlet of the drinking water treatment plants in Louisiana (JP1 in Table 6) and Ontario (WO1 in Table 7) contained naproxen at concentrations ranging from 63 to 65 ng/l. Samples collected at the precipitator of the Louisiana plant (JP2 in Table 6) exhibited naproxen concentrations of 63–68 ng/l, which indicates that the conventional treatment processes and 2-mg/l PAC addition do not remove naproxen from Mississippi River water. Adams et al. (2002) reported no significant removal of selected antibiotics with alum or ferric salt coagulation. Similarly, Ternes et al. (2002) reported no significant elimination of selected pharmaceuticals using iron chloride coagulation. Adams et al. (2002) also reported 25–50% removal of antibiotics from Missouri River water in batch experiments with a PAC dosage of 5 mg/l, and >90% removal for a PAC dosage of 50 mg/l. For the Louisiana drinking-water treatment plant, routine addition of 2 mg/l of PAC, which is used for the removal of natural organic matter in Mississippi River water, does not appear effective in reducing low-level concentrations of naproxen.

Samples collected after chlorination at the Louisiana drinking water treatment plant (JP3 in Table 6) exhibited non-detectable concentrations of naproxen and all other targeted compounds prior to discharge into the distribution system. A sample collected at the Ontario water plant following ozonation, conventional treatment and chlorination (WO2 in Table 7) exhibited non-detectable concentrations of all the target PPCP compounds. Samples collected from the Ontario pilot plant following conventional treatment plus dual media filtration (EN1 in Table 7) and ozonation (EN2 in Table 7) also exhibited non-detectable concentrations for all of the target PPCP compounds. Ternes et al. (2002) reported variable results in reducing concentrations of selected pharmaceuticals using ozone, and Adams et al. (2002) reported reduction of seven spiked (50 µg/l) antibiotics in distilled water and Missouri River water following laboratory chlorination and ozonation. Results from these studies and our results therefore indicate that oxida-

tion (e.g. chlorination and ozonation) and sorption (dual media) processes may be effective treatments for reducing the concentration of naproxen that was observed in Mississippi River and Detroit River waters. Further research is needed to understand the removal processes and the possible formation of byproducts associated with these and other PPCP compounds.

Most of the water samples collected at the Louisiana and Ontario drinking-water treatment plants exhibited non-quantifiable but detectable concentrations of bisphenol A. These observations may be attributed to low-level contamination of the ultra-pure water used for sample preparation in the laboratory, or possible contamination in the plant (Krishnan et al., 1993). More data are therefore needed to determine if containers and/or chemical conveyor systems contribute to low-level bisphenol A contamination in drinking water treatment and distribution systems.

4.4. Application of method

The analytical method developed for this research is suitable for quantitative determination of nine functionally different PPCP compounds from diverse matrices. The method was successfully applied for the analysis of surface waters, wastewater effluent and treated water samples. Application of this method is limited to analysis of the targeted PPCP compounds only. Additional quantities of these compounds could be present in water samples, either in conjugated or other metabolic forms. Further method development would be required to include other chemical forms (e.g. breakdown products or disinfection byproducts) to the list of targeted compounds developed for this study.

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